



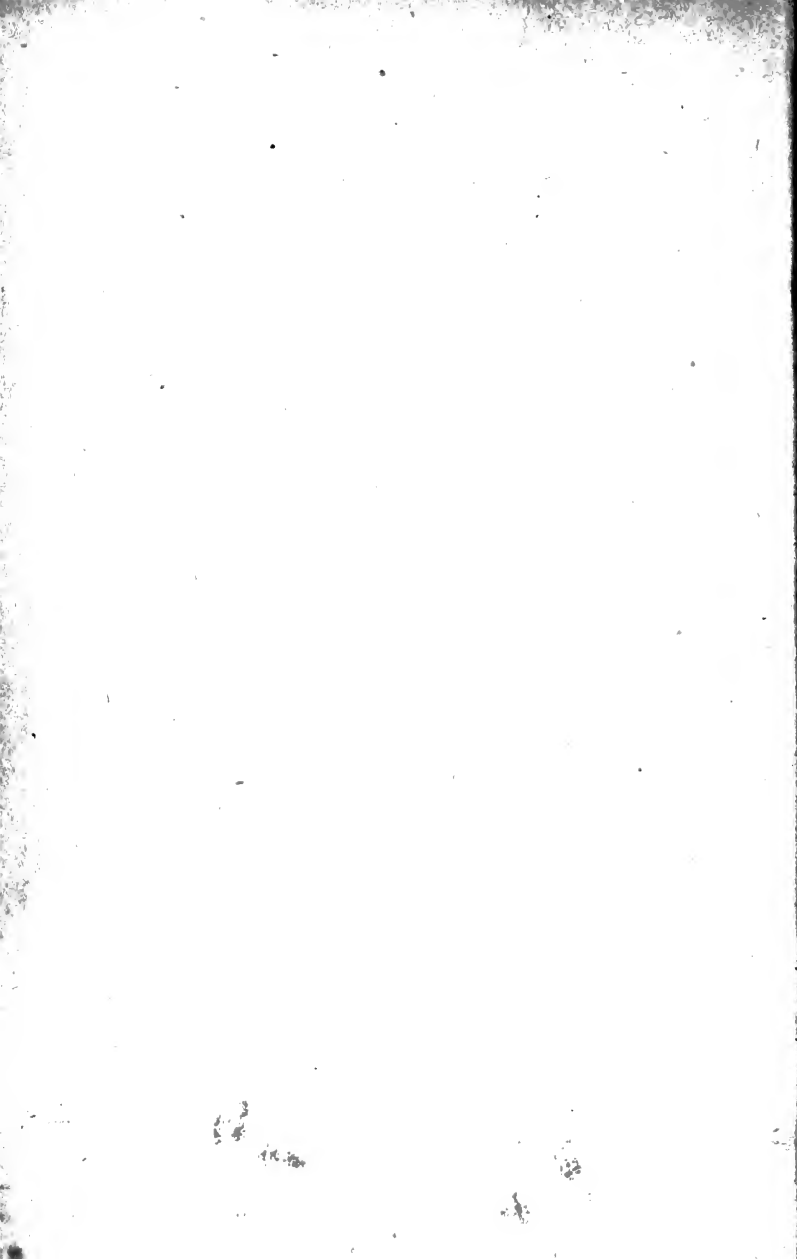
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HANDBOOK
OF
CHEMICAL ANALYSIS
FOR
PRACTICAL MEN.

[CONTAINING
DIRECTIONS FOR EXAMINING AND VALUING SEVERAL HUNDREDS OF THE MOST
IMPORTANT ARTICLES OF COMMERCE, MANUFACTURING PRODUCTS, RESIDUES, ETC.

ALSO,
A BRIEF SYSTEMATIC COURSE OF QUANTITATIVE ANALYSIS,
AND A VARIETY OF USEFUL CHEMICAL TABLES.]

BY
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PROFESSOR OF PRACTICAL AND ANALYTICAL CHEMISTRY.

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P R E F A C E.

THIS little Hand-book is intended to supply manufacturing chemists, dyers, drysalters, druggists, brokers, and all persons interested in the chemical arts, with directions for the assay and valuation of those articles of commerce which come into their hands. For this purpose the best and simplest methods have been selected, and stated, it is hoped, with the needful clearness. The impurities—accidental or intentional—found in each substance are also noted. Articles of food are not included in the plan of the work. To this, the main portion of the manual, has been added a brief course of quantitative analysis, and an Appendix of Chemical Tables. The purpose of the work being merely technological and commercial, many of the niceties and precautions required in abstract research have been necessarily omitted.

The symbol HS has been used for the lengthy words “sulphureted hydrogen.”

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INTRODUCTION.

OPERATIONS IN ANALYSIS.

POUNDING (*pulverization, porphyrization*).—The substance to be examined, if a solid, is reduced to fine powder in a mortar of Berlin ware, or, if very hard, in one of agate. Some minerals can be powdered only if first heated to redness and plunged suddenly into cold water. Tough substances are wrapped up in stout paper, and bruised with a hammer before being placed in the mortar. Metals, alloys, &c., are reduced to powder with a well-tempered and clean file, or else beaten out fine and clipped into shreds with a shears.

DRYING.—Many bodies are capable of absorbing moisture from the air to a large extent. To expel this the powdered substance is exposed to a gentle heat in a small capsule on the sand or water bath, or in the hot-air bath. The temperature should be regulated by a thermometer if sand or hot air be used, and should not, as a general rule, exceed 212° Fahr. Very volatile substances are dried without heat over sulphuric acid. The substance is placed in a capsule or upon a sheet of paper resting upon a wire trellis over a shallow dish containing sulphuric acid. The whole is then set upon a circular block of wood and covered with a bell glass, whose edges fit into a groove cut into the surface of

the wood. This groove being filled with mercury, the interior is completely isolated from the external atmosphere. An exposure of 4 to 12 hours in this apparatus is generally sufficient to remove any trace of moisture.

Sometimes it is preferable to place the substance under the receiver of an air-pump, along with a shallow dish of sulphuric acid, and exhaust the air.

WEIGHING.—The body to be analyzed should be weighed on a counterpoised piece of smooth paper, or in a tared boat or capsule. From 5 to 30 grains may be taken, according to circumstances. No substance should be weighed whilst warm. The balance employed should turn with $\frac{1}{1000}$ th to $\frac{1}{10000}$ th of a grain.

As the amount of hygroscopic moisture has often to be accurately determined, it is sometimes needful to weigh the sample both before and after the desiccation. Such bodies as very rapidly reabsorb moisture may be dried in a small boat of porcelain or platinum. This, with its contents, when dry is inserted into a tube sealed at one end and fitted with a good cork at the other, also accurately tared, and the whole weighed together.

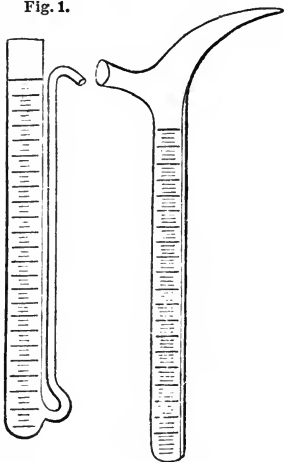
SOLUTION ; DIGESTION.—The ordinary solvents are water, nitric and muriatic acids (singly or together), and sometimes alcohol and ether. Sulphuric acid is rarely applied. The operation may take place in a platinum or porcelain dish, in a glass beaker, or a flask. The latter vessel is preferable whenever a substance has to be submitted to the prolonged action of a volatile acid or of alcohol. Heat is applied by means of the sand or water bath. Where a fixed

temperature higher than 212° is required for digestion, it may be given by means of solution baths and by the oil-bath up to 662° Fahr. Care must be taken that no loss is sustained from spirting or from too violent boiling. To prevent loss of this kind the flasks may be placed in a slanting position. When a substance has to undergo the action of a volatile acid at a high temperature, the escape of the fumes may be much diminished by the following arrangement: Place a clean funnel in the mouth of the flask, and upon it set a porcelain capsule full of cold water. The fumes condense on the bottom of the capsule and trickle back through the funnel. The funnel and the outside of the capsule must be afterwards carefully rinsed into the flask. Some substances can only be dissolved in acids by being first ignited along with caustic soda or lime.

It is often desirable to divide a solution into two or more parts of known amount. This is effected by means of a graduated pourette. Fig. 1 represents Gay Lussac's, and fig. 2 Binks' pourette. In both the liquid is poured in at the large aperture, its bulk noted on the scale of

Fig. 2.

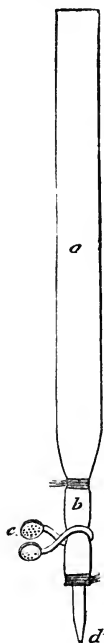
Fig. 1.



degrees, and a known quantity dropped out from the fine aperture.

A still more convenient instrument is Mohr's pourette or alkalimeter, fig. 3; *a* is a tube open at

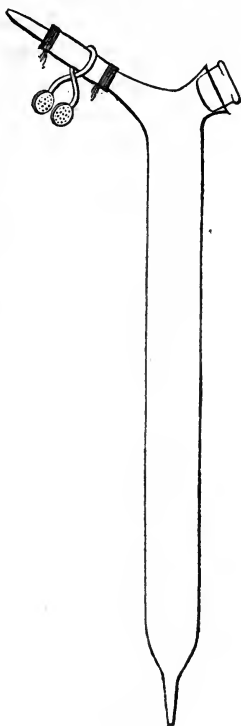
Fig. 3.



the top and drawn out narrower at the bottom, of sufficient size that the graduated part may hold 1000 grain measures of water; *b* is a piece of vulcanized india-rubber tubing which fits tightly on the narrow end of *a*, and may be tied on with a piece of waxed thread so as to be perfectly air tight; *c* is a spring clip, generally made of German silver, which compresses the india-rubber tube so that no liquid can pass. The pressure of this clip may be totally or partially taken off by pressing the two milled heads with the thumb and forefinger, when the liquid trickles out in drops from *d*, a piece of tubing drawn to a fine point, and fitted with wax thread to the end of *b*. The flow of the liquid can be instantly stopped by ceasing to press the milled heads. The whole instrument is suspended from a retort stand or from the wall of the laboratory. This pourette is still open to some objections. The spring clip is damaged by fumes and spirting from the vessel into which the drops fall; the vulcanized tube is attacked by some liquids, *e.g.*, permanganate of potash, and retains a variable quantity of liquid below the clip. To meet these objections I proposed in the *Chemical*

Gazette (vol. xvi. p. 255) the modification shown in fig. 4: *a* is the graduated tube drawn out to a fine orifice, *b*, below, and divided above into two branches. One of these is secured with a ground glass stopper, *c*, whilst to the other are fitted a vulcanized tube, *d*, spring clip, *f*, and capillary tube, *e*. To fill the instrument, take out the stopper, *c*, close the point, *b*, with the finger, and pour in the liquid; replace the stopper and withdraw the finger. No liquid can issue from *b* until air is admitted above by pressing on the mill-heads.

Fig. 4.



Pourettes should always be chosen tall and of narrow calibre, that the degrees may have great perpendicular extent, so that fractions of a degree may be more readily estimated.

PRECIPITATION.—This process is usually performed in glass jars or beakers. The reagent causing the precipitation should in most cases be added in excess. To find whether enough

has been used, take up with a glass rod a drop of the clear liquid which floats above the precipitates, place it in a watch-glass, and test if any portion of the body to be precipitated remain. Some precipitates being subject to change from the action of the air should be filtered as quickly as possible, funnel and beaker being meantime covered with glass plates. Others require to stand for several hours, or to be heated to boiling before they are capable of filtration.

FILTRATION AND WASHING.—The precipitate is separated from the clear liquid by means of a filter. This is a circular piece of filter paper folded first in halves forming a semicircle, and then again so as to form a quadrant. It now will fit in a funnel of proper size. The paper must not reach quite up to the edge of the funnel. The contents of the beaker are now poured into the filter as follows: rub some clean tallow along the *outside* edge of the beaker at the part over which you intend to pour, or along the lip, if such there be. Be careful not to grease the *inside* margin, nor leave any loose particles of tallow. Then proceed to pour, holding a glass rod against the greased edge to guide the stream. The current should be directed not into the apex of the filter (which may previously be moistened with distilled water), but against that part of the side where the paper lies doubled. Any particles of the precipitate which cling to the glass are rinsed into the filter with a current of water from the washing bottle.* This is a flask or

* Precipitates of magnesia especially are apt to cling obstinately to the sides of the beaker, where these have been rubbed with the stirring-rod.

bottle fitted with a cork pierced with two holes. Through one of these passes a glass tube reaching to the bottom of the bottle, bent at right angles, and its outer limb drawn out to a fine point. The other tube merely enters the bottle, and is bent for convenience in blowing. The flask is then nearly filled with water, when by blowing air in at the shorter tube a fine but forcible jet of water issues from the other. This stream is directed chiefly against the upper edge of the filter. The washing liquid is generally distilled water, which may in most cases be hot, but sometimes alcohol or dilute ammonia. Shier's apparatus washes precipitates by means of a current of steam introduced through a bent glass tube passing through a hole in a glass or porcelain plate with which the funnel is covered.

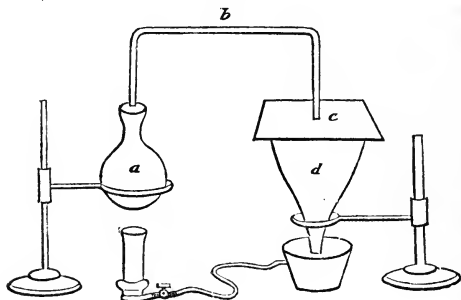
See fig. 5: *a* is a flask containing water; *b* a glass tube bent twice at right angles; the one limb is passed through a perforated cork which fits air-tight in the neck of *a*, whilst the other limb passes through a hole in the plate, *c*, into the funnel, *d*. When the water in *a* is made to boil by a gas jet or spirit lamp, a current of steam passes over into *d* and washes the precipitate on the filter. Care must be taken that the condensed water in *d* do not rise up to the top of the filter.

The washing, in whatever manner conducted, should in most cases be continued till a drop of the liquid

This should, therefore, be avoided as much as possible. Any portion of precipitate, which cannot be removed by washing is, if possible, dissolved in a drop of muriatic acid, again thrown down and added to the bulk.

issuing from the funnel leave no residue when evaporated to dryness on a slip of platinum foil. Precipitates partially soluble in water form, of course, an exception. When the action of air might prove

Fig. 5.



injurious, the funnel is kept covered with a piece of plate glass, or the washing is performed by means of Shier's apparatus. Dr. Normandy supports the filter in a funnel-shaped basket or net of stout platinum wire, which is suspended from the ring of a retort stand. This arrangement allows filtration to proceed with much greater rapidity.

IGNITION.—As soon as the process of washing is completed, the funnel with its contents is placed in the hot-air bath, over the sand-bath, or in some other situation where it may be thoroughly dried, care being taken that no dust fall into the filter. For this purpose it is covered during drying with a piece of porous paper. The ignition takes place in a crucible of platinum, or, in certain cases, of porcelain. The latter material is

used if the precipitate be capable at high temperatures of combining with or otherwise injuring platinum, such as compounds of silver (oxide of lead, tin, antimony, bismuth, cobalt and nickel if a white heat be applied, or if carbon be present), metallic lead, tin, bismuth, phosphorus, phosphoric acid and phosphates in presence of organic matter, alkaline nitrates, alkaline sulphurets, sulphates in presence of carbon and caustic alkalies. The weight of the crucible must be previously ascertained. We place the crucible upon a sheet of highly glazed and coloured paper, and transfer into it by means of a platinum or ivory spatula as much of the precipitate as can be detached from the filter. The filter is then folded up wrapped round with fine platinum wire, suspended by a pair of forceps over the crucible, and set on fire. The ashes are allowed to fall into the crucible, into which are also put any fragments that may have fallen upon the sheet of glazed paper. The crucible is then set over an argand gas-burner or in a gas furnace, and brought to full redness. Of gas furnaces there are several modifications. The commonest is a cylinder of sheet iron 10 or 12 inches high, and 3 or 4 in diameter. The top is covered with fine wire gauze. Gas is introduced by a jet at the bottom, and mingling with common air burns *above* the gauze with a pale blue smokeless flame, in which the crucible is supported in a "crucible-jacket." Another cylinder may be added above the wire to increase the draft. The gas lamps of Hart, Normandy, Solly, and Hoffman, are likewise useful for igniting crucibles. Where gas is not available

the argand spirit lamps of Rose or Berzelius should be employed. If a charcoal furnace is used, a Hessian or other fire-clay crucible is filled with magnesia in which the platinum crucible is bedded.

When perfectly cold, the crucible is weighed along with its contents. A deduction must be made for the ash of the filter. To ascertain its amount, take three of the filters, which should be exactly equal in size, burn them to ashes, weigh the residue, and divide it by three.

If the precipitate be volatile or destructible at a read heat, it is carefully dried at 212° Fahr. as long as any change of weight is perceived, and weighed—a filter of equal size dried at the same heat being used in counterpoise.

In some cases, instead of drying and igniting, it is found preferable to wash the precipitate by decantation, and then introduce it into a Rham's specific gravity

Fig. 6. bottle, fig. 6, which is then filled up to a level with the edge with pure water, and weighed. The difference between the weight thus found and that of the same bottle filled merely with distilled water shows the weight of the precipitate. This method



is particularly applicable to such bodies as chloride of silver, which are very easily reduced if ignited along with any particle of organic matter.—*Mené*.

It is often needful to heat substances in a current of some particular gas, such as hydrogen, chlorine, &c. For this purpose the body to be acted upon is either placed in a small tray or boat of platinum or porcelain, which is then inserted in a glass or porcelain tube, or

a *reduction tube*, fig. 7, is employed, straight or bent, in the bulb of which the substance to be acted upon

Fig. 7.



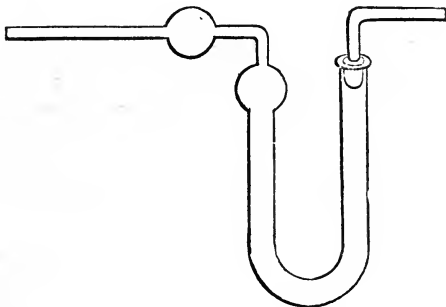
is placed. The gases employed for this purpose are dried by passing them through desiccating tubes, figs.

Fig. 8.



8 and 9, loosely filled with chloride of calcium, or, what is in some cases preferable, asbestos soaked in

Fig. 9.



concentrated sulphuric acid. The tubes are joined together with pieces of vulcanized india-rubber tubing,

or if corrosive gases, such as chlorine, are employed, the ends of the narrower tubes are passed through a perforated cork, which fits exactly into the opening.

When dry substances have to be heated or calcined at fixed temperatures below redness, this is effected by the oil or solution bath, or in the hot-air bath.

Tubes which require heating are passed through a pair of small holes in the opposite sides of a furnace, or a gas or spirit lamp is applied, or by preference are submitted to the action of Hoffman's atmopyre gas furnace for organic analysis, an apparatus which must certainly supersede all other arrangements for heating tubes wherever gas is accessible. Griffin's gas furnace is strongly recommended for almost all purposes for which coke and charcoal furnaces were formerly employed.

CALCULATION OF RESULTS.—We rarely obtain the substance whose amount we are seeking in a separate form, but are obliged to deduce its weight from that of some known compound. Thus, in determining chlorine, we produce and weigh chloride of silver, and find the amount of chlorine by the following proportion:—

$$\left. \begin{array}{l} \text{Equivalent of} \\ \text{chloride of silver,} \end{array} \right\} : \left. \begin{array}{l} \text{Equiv. of} \\ \text{chlorine,} \end{array} \right\} = \left. \begin{array}{l} \text{weight} \\ \text{found,} \end{array} \right\} : x,$$

—the amount of chlorine sought.

PART I.

GENERAL METHODS USED IN QUANTITATIVE ANALYSIS.

§ 1. POTASH AND SODA.

a. The direct separation of these alkalies may be most readily effected by converting them into chlorides.* The mass is then dried, gently ignited in a platinum crucible to expel free acid, and weighed. It is then slightly moistened with water, and treated with an excess—nearly four parts—of crystallized sodio-chloride of platinum, dissolved in the smallest possible quantity of water; the whole is evaporated almost to dryness on the water-bath, in a platinum capsule. The residue is digested for some hours in alcohol of about sixty per cent., collected on a counterpoised filter, washed with alcohol as above, dried at 212° Fahr., and weighed. The drying and weighing should be repeated until the weight proves constant. One hundred parts of the double chloride of platinum and potassium, thus obtained, represent 30.52 parts chloride of potassium.

The chloride of sodium is ascertained by deducting the chloride of potassium thus found from the weight of the mixed salts.

* This may be effected by digesting the substance with an excess of muriatic acid, and evaporating to dryness, except the sulphuric or some other acid which cannot be expelled by muriatic is present.

b. Or, if the mixed salts of potash and soda are chlorides, add a solution of perchlorate of silver in decided excess. Heat the whole nearly to boiling, to allow the chloride of silver to subside; filter and wash with hot water. The precipitate is dried, detached from the paper, and placed in a porcelain crucible; the paper is burnt separately over the lid of a platinum crucible, and the ash is added to the precipitate, which is then ignited and washed. If any black specks remain in the ash of the filter, it is well, before ignition, to moisten with a drop of strong aqua regia, heating afterwards, very gently at first. This process must be repeated as long as any trace of organic matter remains.

From the weight of the precipitate, the *chlorine* present in the mixed salts is known; 143·4 parts of chloride of silver represent 35·4 of chlorine.

The filtrate and washings are now evaporated to dryness, and the residue digested with alcohol. Filter, wash in alcohol, dry, and ignite. The residue thus obtained is *chloride of potassium*.

The alcoholic solution, with the washings, is next evaporated to dryness; the residue ignited in a porcelain crucible and washed with hot water. The washings, when cautiously evaporated to dryness and gently ignited, yield the *chloride of sodium*.

§ 2. If the alkalies are present as sulphates, an excess of perchlorate of baryta is added to the solution. The liquid is heated, allowed to settle—which requires some hours, and is promoted by the addition of a little salammoniac—filtered, the precipitate

washed with hot water, dried, ignited in a platinum crucible, digested afresh in a little dilute hydrochloric acid, filtered, washed, dried, and reignited. It is then weighed, 116.6 parts of the precipitate representing 40.0 of sulphuric acid.

The filtrate is next evaporated to dryness, the residue extracted with alcohol, and the perchlorate of potash remaining insoluble is converted into chloride of potassium, as in I., § 1, *b*.

Note.—In determining sulphuric acid, the liquid must always be acidified by muriatic or nitric acid. The sulphate of baryta is insoluble in muriatic, nitric, and nitro-muriatic acids. Except in very delicate operations, the precipitate of sulphate of baryta may be weighed after one ignition without the subsequent digestion in hydrochloric acid and reignition.

§ 3. The amounts of potash and soda in a mixture of these alkalies may be determined indirectly as follows:—

The alkalies being in the state of chloride—

1. Weigh the mixture.
2. Dissolve, add nitric acid and nitrate of silver in excess, proceed as in § 1, *a*, and weigh the chloride of silver.
3. Multiply the weight, No. 1, by 1.92404.
4. Subtract the product, No. 3, from the weight, No. 2.
5. Divide the remainder, No. 4, by 0.52201.
6. The quotient, No. 5, is the weight of chloride of sodium in the mixture.
7. Subtract No. 6 from No. 1 for the chloride of potassium.

If the alkalies are sulphates—

1. Weigh the mixture.
2. Dissolve, add muriatic acid and chloride of barium in excess; proceed as in § 3, and weigh the sulphate of baryta.
3. Multiply No. 1 by 1.33633.

4. Subtract No. 3 from No. 2.
5. Divide the remainder by 0·29814.
6. The quotient gives the sulphate of soda in the mixture.
7. Subtract No. 6 from No. 1 for the sulphate of potash.

§ 4. LITHIA FROM POTASH AND SODA.

Lithia, when no other base is present, may be weighed as sulphate. For this purpose an excess of sulphuric acid is added to the salt of lithia, and heat applied to expel any other acid present as well as the excess of sulphuric; finally apply a red heat, and weigh the residue when cool.

If potash and soda are also present, the whole are, if possible, converted into chlorides, and the weight of the mixed salts ascertained. The potash is then determined, as in I. § 1, by means of chloride of platinum. The filtrate, after removal of the precipitate of potash-chloride of platinum, is evaporated to dryness and ignited; the residue is dissolved in water and filtered, to get rid of metallic platinum. To the clear liquid some pure phosphoric acid is added, and an excess of pure carbonate of soda. The whole is evaporated to dryness, treated with cold water, thrown on a filter, washed with a little cold water, dried, ignited, and weighed. It contains 12·32 per cent. of lithia. The amount of chloride of potassium found is added to the quantity of chloride of lithium equivalent to the lithia obtained (14·4 parts of lithia = 41·8 parts chloride of lithium), and the sum subtracted from the original weight of the mixed chlorides. The remainder is chloride of sodium.

Mixed chlorides of lithium and sodium may be

treated with a mixture of absolute alcohol and anhydrous ether, which dissolves out the former. The liquid is filtered, the solid matter washed with alcohol and ether, dried, ignited, and weighed as chloride of sodium. The solution and washings are evaporated to dryness and gently ignited, and weighed as chloride of lithium.

§ 5. SEPARATION OF CAUSTIC ALKALIES FROM THEIR
CARBONATES.

Dissolve in water, filter, and add to the clear solution chloride of barium in excess. Filter off the carbonate of baryta, wash, dry, ignite in a platinum crucible, and weigh. 98·6 parts of the precipitate represent 69·2 parts carbonate of potash, or 53·2 carbonate of soda. The filtrate is next treated with a current of carbonic acid gas. The precipitate produced is filtered off, &c., as above. 98·6 parts of this second precipitate represent 47·2 of potash, or 56·2 of the hydrate of potash, or 30·97 of soda, or 39·97 hydrate of soda. Instead of precipitating with carbonic acid, the amount of caustic alkali in the filtrate may be determined by adding a standard acid. (See II., § 1.)

§ 6. ALKALINE SULPHATES AND SULPHURETS.

A known quantity, *e.g.*, 10 grs. of the substance should be dissolved in water, and strongly acidulated with hydrochloric acid. Filter if any turbidity appears. To the clear liquid add chloride of barium, and determine the sulphuric acid, as in I., § 2, from

which the amount of sulphate of potash or of soda is deduced.

Take another portion of the substance and digest it with a strong solution of the permanganate of potash in excess; when the latter no longer suffers decomposition, but retains its purple tint, add muriatic acid in excess, filter, and determine the sulphuric acid as before. If we subtract the first quantity of sulphuric acid from the second, every 10 parts in the remainder answers to 4 of sulphur present in the form of alkaline sulphuret.

In this manner determine sulphur in alkaline and earthy sulphurets.

§ 7. SULPHURETS, SULPHITES, HYPOSULPHITES, AND
SULPHATES OF ALKALI.

Add to the mixed solution carbonate of cadmium, and digest for some time with frequent stirring. Filter off the mixture of sulphuret and undecomposed carbonate of cadmium, and treat the precipitate with acetic acid to remove the latter salt. The remaining sulphuret of cadmium is treated with nitric acid and filtered. The undissolved portion is collected on a balanced filter, dried, and weighed as sulphur. The nitric acid solution is treated with nitrate of baryta, which throws down sulphate of baryta. This is collected on a filter, &c., and weighed. Its amount of sulphur, together with that weighed in a separate form, give the total of sulphur present in the mixture in the form of an alkaline sulphuret. To the clear liquid filtered off from the salts of cadmium nitrate of

silver is added, as long as anything is thrown down. The mixture is kept for some time at a temperature near ebullition. The precipitate (sulphuret of silver) is then collected, washed, partially dried, and treated with strong nitric acid, and the sulphuric acid thus formed determined in the usual manner. 106·6 parts of the sulphate of baryta obtained here represent 24 parts of hyposulphurous acid.

The sulphuric acid in the filtrate from the silver is next determined as above, from which we deduct the sulphuric acid found in a fresh portion of the original solution, previously rendered acid by the addition of muriatic acid. The remainder shows the amount of sulphuric acid representing the sulphurous acid of the mixture.—*Kynaston*.

§ 8. CARBONIC ACID IN ALKALINE CARBONATES.

a. Treat carefully with pure dilute hydrochloric acid until all effervescence ceases. Evaporate to dryness, and heat until the last traces of free acid are expelled, *i. e.*, until a moistened piece of litmus paper, suspended over the capsule, is no longer reddened; but avoid such an excess of temperature as might volatilize any portion of the chloride. Dissolve the residue in water, and therein determine the chlorine—§ 3, 2. This will be exactly proportional to the carbonic acid originally present, 35 parts of the former representing 22 of the latter.

In like manner determine all bodies capable of being readily, and without loss, converted into chlorides, as—

1. *Earthy carbonates* are mixed with muriatic acid

in excess, evaporated to dryness, redissolved in water, filtered to remove any silica, &c., and the chlorine in the clear liquid is determined as usual.

2. *Vegetable salts of earths and alkalies* (e. g., acetates, malates, citrates).—Convert into carbonates by calcination, and treat as above.

3. *Chlorates of the alkalies* are converted into chlorides by careful calcination, or by heating with *pure* peroxide of manganese. The chlorine is then determined.

4. *Perchlorates* may be treated in the same way.

5. *Neutral nitrates* are converted into chlorides by boiling with an excess of hydrochloric acid.

6. *Sulphites* are also treated in the same manner, taking care to expel all traces of the sulphurous acid by prolonged heating. In all these cases where one base only is combined with the acid in question, its amount may readily be found by calculation.

7. *Carbonic acid* (as evolved from the decomposition of a carbonate by an acid, or from the combustion of an organic body) is absorbed by a mixture of chloride of barium and ammonia. The whole is boiled, filtered, and the precipitate washed until the liquid running through no longer renders nitrate of silver turbid, or turns red litmus paper blue. The precipitate (carbonate of baryta) is dissolved in dilute muriatic acid, evaporated to dryness, redissolved in pure water, and the chlorine in the perfectly neutral solution determined by nitrate of silver.

8. *Ammonia* is saturated with muriatic acid, evaporated to dryness—avoiding all excess of temperature

—and the chlorine in the perfectly neutral residue determined as usual.

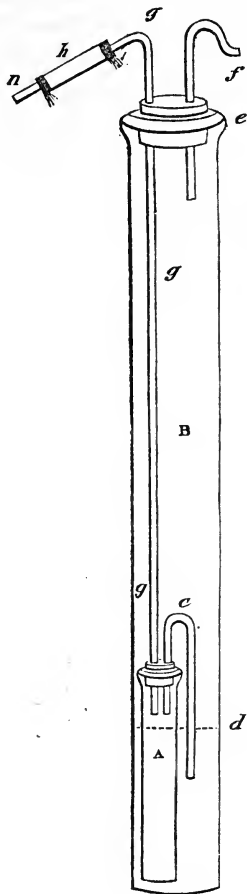
This process is inapplicable in the case of bodies whose chlorides are volatile or unstable. It cannot be extended to mixtures of neutral with sesqui- or bi-carbonates, nor, generally speaking, to subsalts.

Chlorides already existing along with any of the above-mentioned bodies must, of course, be previously determined.

b. The carbonic acid in alkaline carbonates may also be determined as follows:—

From 20 to 100 grains of the substance are placed in a test-tube (fig. 10), and moistened with a little water. This tube, which we will call A,

Fig. 10.



is fitted with a good cork, pierced with two holes. Through one of these passes a piece of fine glass tubing, *c*, which is bent downwards just outside the cork, and runs parallel with it for about two-thirds of its length. Through the other hole passes a similar tube of considerable length, *g g*, rising straight upwards. Neither of these tubes should enter A to any depth, but merely pass through the cork. The whole is then placed in a much larger test-tube, B, which contains concentrated sulphuric acid up to the dotted line, *d*, and into which the downward fine tube should dip for about an inch. B is also fitted with a good cork, *e*, pierced with two holes. Through one of these is passed a short curved piece of tubing, *f*, whilst through the other rises the upright tube, *g g*, from A. To the extremity of this upright a piece of caoutchouc tubing, exactly fitting, is tightly tied, and temporarily closed, by inserting a piece of glass rod, *n*. When the apparatus is perfectly air-tight, it is weighed on a delicate balance, and the weight noted down. A little air is then sucked out of the apparatus by applying the lips, *dry*, to the end of *f*, whereon a drop of the sulphuric acid in B rushes up into A, and liberates carbonic acid gas, which escapes through *c*, and is perfectly dried by the sulphuric acid through which it has to pass. When the effervescence has ceased, suction is again applied, to cause another drop of sulphuric acid to enter A. This is continued until the sample is perfectly decomposed, taking care never to allow much acid to enter A at once. When all effervescence has ceased, a further

quantity of acid is sucked into A, that by the consequent rise of temperature the last traces of carbonic acid may be expelled. The piece of glass rod is next taken out of the caoutchouc tubing, and by again applying the lips to the curved tube, a stream of air is drawn through the apparatus. In cases of great nicety, a glass tube containing chloride of calcium, or asbestos moistened with sulphuric acid, is affixed to the caoutchouc tube, in order that the atmospheric air drawn in may be perfectly freed from moisture. The entire apparatus is lastly weighed, when the loss shows the quantity of carbonic acid originally present in the sample.

Care must be taken that the tube A be sufficiently capacious. The piece of glass rod must, of course, be replaced before the final weighing.

§ 9. MAGNESIA AND ALKALIES.

a. Dissolve the substance in nitric acid, evaporate to dryness; add water, and an excess of pure oxalic acid. Evaporate to dryness at a boiling heat again, and apply heat sufficient to decompose the oxalates. The residue is extracted with boiling water, kept for some time in a state of ebullition, filtered and washed with boiling water, or by means of Shier's apparatus. The precipitate is then dried, ignited, and weighed as *pure magnesia*. The ignition must be somewhat prolonged, to expel the last traces of carbonic acid. Lime must not be present. The alkalies are found in the filtrate, and may be estimated as directed above; or—

b. Add to the solution—which must contain neither

lime nor ammoniacal salts (if the latter are present, evaporate to dryness, and apply a red heat for their expulsion, then redissolve in water with a little nitric acid, if necessary)—lime water, until it has a distinctly basic reaction. Boil and filter off the precipitate of magnesia, as above. The precipitate when dried, ignited, and weighed, may be redissolved in a hot concentrated solution of nitrate of ammonia, when any traces of lime which may have been thrown down will remain undissolved, and their weight must be deducted from the amount previously found.

The filtrate containing the alkalies is poured into an excess of carbonate of ammonia, the precipitate filtered off, and washed with boiling water. The alkalies are determined in the clear liquid; or—

c. Mix the solution with muriate of ammonia, boil down to dryness, redissolve in a little water, and add saturated baryta water. Magnesia is thrown down, filtered off, &c., and weighed. The filtrate is freed from baryta by sulphuric acid, filtered, the clear liquid evaporated down, and ignited with a little carbonate of ammonia. The alkalies remain as sulphates; or—

d. Add to the solution sal-ammoniac, free ammonia, and phosphate of ammonia. The magnesia is thrown down as double phosphate of magnesia and ammonia, which is treated as in I. § 9, *g*. It contains 35.9 per cent. of magnesia.

The filtrate and washings are mixed; they contain the alkalies, along with ammoniacal salts and excess of phosphoric acid. Evaporate to dryness in a platinum capsule, ignite to expel ammonia; take up again in

water, add nitrate of silver and carbonate of silver in excess. When the precipitate is collected, and the liquid clear and quite neutral, it is filtered off. To the filtrate is added hydrochloric acid in excess, to remove the residue of silver. Filter again, and in the filtrate determine the alkalies which are quite free from any other fixed matter; or—

e. Render the solution as neutral as possible by evaporating away excess of acid. When cold, add an excess of sesquicarbonate of ammonia, and allow to stand for some time. Filter with a solution of carbonate of ammonia. The precipitate of magnesia, after ignition, still retains some carbonate of potash. It is therefore again washed, dried, ignited, and weighed, the washings being added to the original filtrate. The precipitate contains 15.9 per cent. of magnesia (*Schaffgotsch*). This process is available for separating magnesia from lithia; or—

f. The magnesia and alkalies being in the state of chlorides, mix with red oxide of mercury, and boil down to dryness. Redissolve in water, filter, &c., and weigh the well-ignited precipitate as pure magnesia. In the filtrate the alkalies are contained as chlorides.

Solutions containing magnesia should always be evaporated down at a boiling heat, stirring diligently to prevent spirting. At lower temperatures the salts are apt to climb up the sides of the vessels and occasion loss. All the above processes are best performed in platinum vessels.

g. Magnesia is often determined by adding to its

solutions a mixture of phosphate of soda and ammonia. The whole is allowed to stand for about twelve hours in a warm place. The precipitate is filtered, washed with very dilute ammonia, dried, ignited, and weighed as neutral phosphate of magnesia, 100 parts of which represent 35.9 of magnesia; or—

h. Carbonate of potash in excess may be added to the solution, the mixture boiled for a considerable time in a platinum capsule, the precipitate filtered, washed with boiling water, dried, thoroughly ignited, and weighed as pure magnesia.

Salts of ammonia, if present, must be previously destroyed by adding carbonate of potash in very considerable excess and boiling, until on a further addition of carbonate of potash ammonia is no longer given off.

As some magnesia often escapes being thrown down, it is prudent to evaporate the filtrate to dryness in a platinum capsule at a strong heat, stirring continually to prevent spirting. The residue is extracted with boiling water, and any carbonate of magnesia which remains undissolved is filtered off, washed, &c., and its weight added to the amount previously obtained.

Where great exactness is required, the magnesia after weighing is dissolved in dilute hydrochloric acid. A trace of silica (derived from the carbonate of potash) often remains behind. This is filtered off, washed, &c., ignited, and its weight deducted from that of the magnesia as already found; or—

i. Evaporate to dryness in the water-bath the filtrate remaining after precipitation of all the bases.

Ignite the residue cautiously in a platinum crucible, to drive off all ammoniacal salts. All loss may be prevented by using as lid a capsule with the convex surface turned downwards. Moisten the mass with dilute sulphuric acid and again evaporate to dryness, repeating this operation until no other acid is present but the sulphuric. To render the acid salts neutral place a piece of carbonate of ammonia in the crucible, and heat again until the bottom becomes slightly red. Repeat this until the weight becomes constant. The contents of the crucible are now dissolved in water, the sulphuric acid in the solution determined by chloride of barium, the excess of baryta removed by means of sulphuric acid, the magnesia precipitate by ammonia and phosphate of soda, and determined as usual.

From these three data—

The quantity of sulphates = A

(If the whole is not dissolved when treated with water, the weight of the insoluble residue must be deducted from A.)

The quantity of sulphuric acid contained in them..... = S

The quantity of magnesia in them..... = m

the amount of potash and soda is calculated. If by S^1 is represented the sulphuric acid combined with m , and by s the sulphuric acid combined with potash and soda, by a the quantity of alkaline sulphates contained in it, then—

$$a = A - (m + S^1), \text{ and } s = S - S^1.$$

The quantity of potash, KO, contained in the mixture is shown by the formula—

$$x = \frac{\frac{s \cdot \text{NaO}}{\text{SO}^3} + s - a}{\frac{\text{NaO}}{\text{KO}} - 1}; \text{ or } x = \frac{1.77829 \cdot s - a}{-0.33816}$$

$$y = \frac{\frac{s \cdot \text{KO}}{\text{SO}^3} + s - a}{\frac{\text{KO}}{\text{NaO}} - 1}; \text{ or } y = \frac{2.17595 \cdot s - a}{0.5109}$$

x and y denote respectively the quantities of potash and soda sought for. NaO, KO, and SO³, represent the atomic weights of soda, potash, and sulphuric acid.

§ 10. LIME FROM MAGNESIA.

Dissolve in dilute nitric or hydrochloric acid, and render the solution slightly alkaline by means of ammonia. Add oxalate of ammonia, allow the mixture to stand for eight or nine hours without applying heat, filter, wash the precipitate dry, ignite till no further change of weight occurs, and weigh as *lime*.

Evaporate the filtrate to dryness with an excess of nitric acid to destroy ammoniacal salts; redissolve the residue in water with a little nitric acid. For the determination of the magnesia and alkalies see I. § 9.

If it is desired to separate lime from magnesia in presence of phosphoric acid, the substance is dissolved in a very little hydrochloric acid. Ammonia is added until a permanent precipitate appears, which is redissolved in acetic acid. Oxalate of ammonia is next

added in excess, and lastly acetate of soda. The precipitate of oxalate of lime is treated as usual.

Lime in presence of alkalies, no other earth being present, is determined by means of oxalate of ammonia as above. The filtrate is evaporated to dryness and heated to destroy the ammoniacal salts, and the alkalies are then estimated in the residue as in I. § 1.

§ 11. BARYTA FROM LIME.

Dissolve in nitric or muriatic acid; dilute with water and add a sufficiency of sulphuric acid. The precipitate is treated as directed in I. § 2, and the amount of baryta calculated from the sulphate of baryta weighed.

The lime is determined in the filtrate as in I. § 10.

§ 12. STRONTIA FROM LIME.

Precipitate both as carbonates; wash well in hot water, and redissolve in nitric acid, applying a gentle heat, and taking care not to use any more acid than is absolutely needful. Evaporate to dryness in a flask provided with a stopper, which must be inserted as soon as the residue is dry. When the mass is cool, pour into the flask absolute alcohol (about twice the bulk of the residue), replace the stopper, and allow the flask to stand in a cool place for some time with occasional shaking. Nitrate of lime dissolves, whilst nitrate of strontia remains insoluble. The whole is thrown upon a balanced filter and washed with anhydrous alcohol, the funnel being kept covered with a

glass plate during filtration. The filter, with its contents, is dried and weighed.

The filtrate is mixed with water, heated to expel alcohol, and the lime determined as usual.

§ 13. BARYTA FROM STRONTIA AND LIME.

Dissolve in excess of hydrochloric acid; add hydro-fluo-silicic acid and allow the whole to stand for some time. Collect on a weighed filter, wash, dry at a gentle heat, and weigh; 100 parts contain 49.10 of barium.

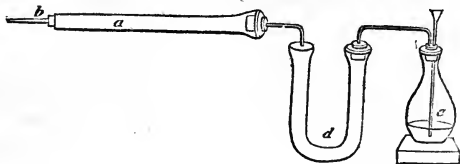
The filtrate is mixed with sulphuric acid evaporated to dryness, and the residue ignited. The strontia is calculated from the weight thus found, if no other base is present. If lime occurs along with the strontia, the mixed sulphates are ignited with thrice their weight of carbonate of soda in a platinum crucible. The resulting mass is well washed with water, and treated as in I. § 12. Magnesia or alkalis occurring along with strontia are removed by washing the mixed sulphates with very dilute alcohol. *

§ 14. ALUMINA AND PEROXIDE OF IRON.

a. Dissolve in nitric or nitro-muriatic acid, and heat for some time that all iron may be peroxidized. Precipitate the mixture with ammonia, filter, wash, dry, detach the precipitate from the filter and burn it, adding its ashes to the precipitate which is ignited in a platinum crucible. After weighing it is finely powdered, placed in a balanced porcelain boat, and weighed again. The boat is introduced into a

porcelain tube, fig. 11, *a*, passing horizontally through a Hoffmann's gas furnace. To one end of this tube

Fig. 11.



a drawn-out glass tube, *b*, is attached by means of a good perforated cork. Meantime, a slow current of hydrogen gas is generated in the flask, *c*, perfectly dried by being allowed to pass through the U-tube *d* (which is loosely filled with asbestos moistened in strong sulphuric acid), and allowed to enter *a* at the other end. When the common air is quite expelled from the tube, the furnace is lighted, and the tube raised to low redness about the part where the boat lies, the remainder being merely kept hot enough to prevent the deposit of watery vapour. The heat is kept up as long as water is deposited in the drawn-out glass tube, *b*. The gas is then turned off, and the current of dry hydrogen gas kept up till the apparatus is quite cold. In default of a Hoffman's furnace the tube may be heated in a Luhme's coke furnace. When cold, the boat is taken out of the tube and weighed. The loss shows the oxygen which was in combination with the iron, from which both the alumina and iron may of course be calculated.

By way of confirmation the powder contained in

the boat is digested in the cold for twenty-four hours in very weak nitric acid (1 part acid to 30 or 35 of water), and the solution is filtered. The alumina which remains undissolved is washed, dried, ignited, and weighed.

The filtrate is heated, and mixed with ammonia to precipitate sesquioxide of iron, which is filtered, washed, dried, ignited (burning the filter separately), and weighed—78 parts representing 54 of metallic iron.

b. Alumina when alone, or only accompanied by alkalis (as in alums) is determined by adding carbonate of ammonia (or pure ammonia when carbonic acid would be objectionable), washing with boiling water, drying very gradually and igniting, commencing with a very gentle heat, and weighed as pure alumina.

c. Alumina and iron may also be separated as follows:—The substance is dissolved in muriatic acid and peroxidized by boiling with nitric acid. All superfluous acid having been evaporated off, add neutral tartrate of ammonia and then caustic ammonia until it is decidedly alkaline. Should it grow turbid, more tartrate of ammonia is added until all is perfectly dissolved. Sulphuret of ammonium is then added in excess to the clear solution. The whole is heated on a sand-bath, the precipitate of sulphuret of iron filtered off, and washed with hot water mixed with a little sulphuret of ammonium, and at the commencement with some tartrate of ammonia. It is then dissolved in muriatic acid, peroxidized with

nitric acid, and determined as in I. § 15. The filtrate is evaporated to dryness, ignited in a platinum capsule to expel ammoniacal salts, and weighed as pure alumina.

Chrome, if present, accompanies the alumina. They are separated as in I. § 56.

Alumina may be separated in the same manner from manganese and cobalt.

Alumina is not perfectly precipitated from its solutions by ammonia in presence of organic matter.

Aluminous precipitate are very tedious to wash; to abridge this operation they may be once treated with hot water, then dried, placed together with the filter in a platinum crucible, and heated to dull redness. The mass is now triturated with hot water, and filtered again, dried, ignited, and finally weighed.

§ 15. IRON.

Iron, when unaccompanied by any other base except the alkalies, or lime, baryta, and strontia, is determined by dissolving in water or hydrochloric acid, as the case may be. The solution is then treated with chlorine gas, or digested for some time with nitric acid, to peroxidize all the iron. Ammonia (as free as possible from carbonate) is now added, the solution having been first moderately diluted. It is filtered, washed in hot water, dried, ignited, and weighed as sesquioxide.

§ 16. IRON FROM ZIRCONIA.

Proceed as in I. § 14. The nitric acid may be used stronger.

From *glucina* iron is separated in the same way; but here exceedingly weak nitric must be applied.

The zirconia and glucina in both cases are weighed as such.

§ 17. IRON FROM CHROME.

As in I. § 14; chromic oxide remains undissolved after the iron is dissolved out, and may be weighed as such.

§ 18. PHOSPHORIC ACID.

1. If no earth or metallic oxide be present, add to the solution a mixture of sulphate (or acetate) of magnesia with ammonia and sal-ammoniac. No more of the latter should be added than is necessary to prevent the magnesian salt from being rendered turbid by the ammonia. This mixture is added as long as a precipitate continues to form, and until the liquid smells of ammonia. The whole is allowed to stand in the cold for twelve hours. It is then filtered, washed with dilute ammonia until chloride of barium does not render the washings turbid, dried, ignited, and weighed. 100 parts of the precipitate contain 64.1 of phosphoric acid.

2. Or, take a known weight of the purest metallic iron, dissolve it in hot nitric acid, and add it to the solution in question; then precipitate with an excess of ammonia, filter, wash, dry cautiously, and ignite, commencing with a very gentle heat. The weight of the precipitate *minus* the known amount of peroxide of iron, gives the phosphoric acid. Care must be taken to add a sufficient excess of iron. Every 27 parts of metallic iron give 39 of peroxide.

3. Phosphoric acid may also be determined by one of the following methods, which are available in presence of the alkaline earths, earths, &c., and are therefore used in the analysis of soils, manures, ashes of vegetables, &c.:—

a. Uranium Process.—The substance is dissolved in acetic acid; or, if that be impracticable, in a little nitric or hydrochloric acid; concentrate the solution as far as possible; add acetic acid in excess to redissolve the precipitate, and acetate of ammonia if mineral acids be present. If iron be present, the acetic acid must be added very freely, and the solution boiled for about half an hour. The whole is now set aside, and when somewhat cooled a few drops of chloroform are added, the liquid stirred, boiled up once more, and allowed to settle. When the liquid above the precipitate is clear, it is decanted into the filter as far as possible. Water is added to the residue, which is again boiled, allowed to settle, the clear liquid poured into the filter, and this process repeated three or four times until the precipitate loses its slimy appearance. It is then entirely thrown upon the filter, washed, and dried as usual. The precipitate is now separated from the filter and placed in a large platinum crucible, the filter burned separately, its ashes added to the precipitate, and the whole ignited; when cool it is moistened with nitric acid, dried again, ignited, and weighed. 19.99 per cent., or very nearly 1-5th of the weight, is dry phosphoric acid; or—

The fluid containing the precipitate is evaporated to perfect dryness on the water-bath, the residue

digested for some time in strong acetic acid, mixed then with water heated nearly to boiling, and digested for some time longer. The precipitate is poured as far as possible into the bottom of the filter. If any part of the precipitate is found adhering to the bottom of the vessel in which the evaporation was performed, it is dissolved in a few drops of hydrochloric acid, reprecipitated with ammonia, and so added to the rest.

If organic matter be present, as in urines, manures, &c., and the whole substance cannot be incinerated without decomposition, we add sal-ammoniac and sulphate of magnesia in excess, and then with ammonia precipitate phosphates along with earths, &c. This precipitate is filtered off, dried and burnt without washing, re-dissolved in a little hydrochloric acid, and then treated with acetate of uranium as above.

b. Molybdate of Ammonia Process.—Dissolve the substance in nitric acid. Next dissolve at a gentle heat 2 parts of pure molybdic acid and 1 part of tartaric acid in 15 parts of water. Add 10 parts ammonia of specific gravity 0.97, and 15 parts of nitric acid. Boil the whole in a porcelain vessel, and filter. A sufficient quantity of the test liquor—which should contain at least 30 times as much molybdic acid as there is likely to be phosphoric acid in the substance—is put in a capsule, boiled, and the solution of the phosphate added. The precipitate and liquid are digested together for some hours at a gentle heat, filtered, and the precipitate washed with water containing $\frac{1}{10}$ of nitric acid. The precipitate on the filter is dissolved in ammonia, and the solution treated with a salt of

magnesia as in I. § 18, 1. The filtrate is allowed to stand for some hours in a warm place, to see if anything further is deposited. If this occur, it is filtered off and put in the ammoniacal solution.

Note.—In these processes error may be occasioned by the presence of arsenic or silica. The former, where it occurs, may be removed by passing a current of HS through the acid solution of the substance and filtering. Soluble silica may be removed by evaporating the acid solution of the substance to dryness, and redissolving in a little nitric or hydrochloric acid, when the silica remains insoluble and is filtered off.

c. Nitrate of Bismuth Process.—Prepare a solution of nitrate of bismuth as follows:—Dissolve pure crystallized subnitrate of bismuth in 4 parts nitric acid of specific gravity 1.36, add 30 parts of distilled water, boil, and filter if requisite.

The substance to be examined is weighed out, dissolved if possible in water, otherwise in nitric acid, avoiding excess. Sulphuric and muriatic acid, if present (either free or in the form of soluble chlorides or sulphates), are removed by digestion with a sufficient quantity of the nitrates of baryta and silver, the precipitate formed filtered off, very carefully washed, and the washings added to the original solution. Ammoniacal salts, if present, as in guanos, urines, &c., should be expelled either by carefully heating to low redness, or by boiling with excess of pure carbonate of soda as long as ammoniacal fumes are given off. The substance must then be dissolved in nitric acid as above. The nitrate of bismuth is then added, and the white precipitate, which falls very rapidly, collected on a filter, washed, dried, ignited in a

platinum crucible, and weighed. 23.28 per cent. of the weight thus obtained represents dry phosphoric acid.

In case of meta- and pyro-phosphoric acids, the solution, after addition of the nitrate of bismuth, is boiled for a short time, when the precipitate is rendered similar in composition to that formed with the ordinary phosphates.

§ 19. PHOSPHORIC ACID FROM SILVER, GOLD, BISMUTH,
COPPER, LEAD, ARSENIC, ANTIMONY, ETC.

From all substances whose acid solutions are precipitable by HS , phosphoric acid may be separated by means of that reagent, and afterwards determined by one of the foregoing methods.

Phosphoric Acid combined with Protoxides.—Dissolve a weighed portion of the substance in nitric acid, avoiding too great excess, and dilute with water. Add to the clear liquid, having filtered off any insoluble matter, a sufficiency of nitrate of silver, and then carbonate of silver in small excess. In a short time a yellow precipitate appears. When the liquid no longer reddens litmus, the precipitate is thrown on a filter, washed, and the filter afterwards burst, and the precipitate washed out into a flask, where it is redissolved in nitric acid. The silver is next thrown down by muriatic acid, filtered off, washed, the washings added to the filtrate, and therein the phosphoric acid determined with magnesia and ammonia as above.

§ 20. PHOSPHORIC ACID FROM COBALT, MANGANESE, ZINC,
AND IRON.

Dissolve in muriatic or nitric acid, add ammonia in moderate excess, and afterwards an excess of hydro-sulphate of ammonia, in which the precipitate is digested for some time. The amount of oxide is determined from the sulphurets thrown down, and this, deducted from the original weight of the sample operated upon, gives the amount of phosphoric acid.

§ 21. PHOSPHORIC ACID FROM IRON, NICKEL, OR COBALT.

Powder finely, suspend in caustic potash or soda, and pass a stream of chlorine gas through the liquid. The metal is thrown down as peroxide or sesquioxide, whilst an alkaline phosphate remains in solution, in which the phosphoric acid is determined by one of the above-mentioned methods.

§ 22. PHOSPHORIC ACID FROM IRON AND ALUMINA (SOILS.)

Add to the solution (aqueous or acid, acidulating with hydrochloric acid in the former case) a little perchloride of iron, and acetate of soda in excess. If the liquid does not turn red, more perchloride of iron must be very gradually added until this takes place. Pour it into a flask, and boil for a few minutes. A reddish-brown precipitate falls. If the liquor above this is not colourless, more acetate of soda is added and the boiling repeated. Filter, wash with boiling water, then make a hole in the filter, and wash the moist precipitate into a beaker. Dissolve in hydrochloric acid, add tartaric

acid, and pour in ammonia until the precipitate which is at first formed redissolves. Add acetate of lead in slight excess, filter and wash the precipitate, redissolve in a minimum of nitric acid, and add subacetate of lead in excess. The precipitate is filtered, &c., ignited, and weighed. It is then placed in a beaker, dissolved in dilute nitric acid, with which the crucible is also thoroughly cleansed, and the rinsings added to the solution. The liquid is now freed by evaporation from all excess of acid, and mixed with alcohol and pure rather dilute sulphuric acid in excess. Filter the precipitate after allowing it to stand for some time, wash in dilute alcohol, dry, and ignite in a porcelain crucible, burning the filter separately. From the sulphate of lead thus weighed the amount of *oxide of lead* present is found (151.56 sulphate represent 111.56 oxide), and this amount, deducted from the former weighing, gives the quantity of phosphoric acid.

§ 23. GENERAL METHOD FOR MIXED EARTHS AND ALKALIES.

Separation of alumina, iron, manganese, lime, magnesia, potash, and soda.

Dissolve in nitric acid, evaporate to dryness, and heat gradually upon the sand bath to 392° – 482° Fahr., maintaining the temperature till a glass rod, moistened with ammonia and held over the vessel, no longer shows the presence of acid fumes. This operation should be performed in a platinum dish, provided with a lid, both balanced. Moisten the residue with a concentrated solution of nitrate of ammonia, and heat afresh, repeating this process till

no more ammoniacal fumes are given off. Treat the mass with water, and digest at a gentle heat. Add one drop of weak liquid ammonia, which will cause no turbidity if the heating has been properly conducted.

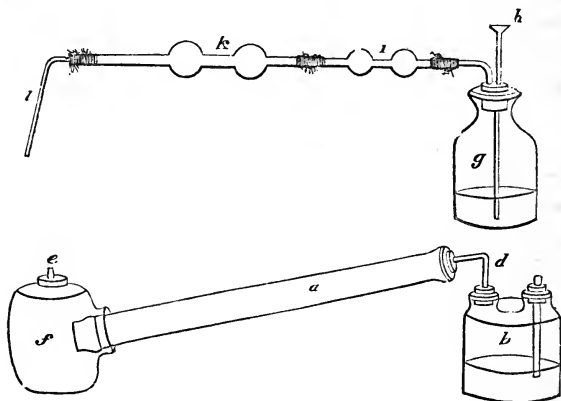
Decant off the liquid portion, A, from the insoluble, B. Digest the latter afresh in water, and wash it in boiling water by decantation, and add the washings to A.

The solid portion, B, containing the alumina, iron, and manganese, when thoroughly washed, is ignited (still in the same platinum crucible) and weighed. It is treated with moderately strong nitric acid at a gentle heat.

Peroxide of manganese remains undissolved, and is washed, dried, and weighed on a balanced filter. *Alumina and sesquioxide of iron*, along with a trace of *manganese*, dissolve, and are thus separated. Add ammonia, dry, ignite, and weigh the mixed precipitate. Powder it finely, and place in a platinum boat, also of known weight, and weigh again to find how much of the material has been lost in pulverizing. This weighing is best performed in a glass tube, closed at one end, fitted with a cork at the other, and previously balanced. The boat is then put in a porcelain tube, *a*—fig. 12—passing through a furnace in a slanting direction. The lower end of the tube enters into a small balloon, *b*, with its tubulure, *c*, turned upwards. Meantime a current of hydrogen gas is generated from pure granulated zinc, and dilute sulphuric acid in the flask, *g*, passed through the bulb-tube,

i, from thence through the tube, *k*, filled with fragments of chloride of calcium, to dry the gas; thence it is allowed to pass into *a* by plunging the down tube *l* into *f*, a piece of wider tubing fixed air-

Fig. 12.



tight, by means of a perforated cork, into one of the necks of *e*, a two-necked bottle containing concentrated sulphuric acid. The tube *f* reaches below the level of the acid, and *l* is plunged in so as to go below the lower end of *f*, and nearly touch the bottom of the bottle. The gas escapes into *a* through *d*, an elbowed tube fixed air-tight into the other neck of *e*, and into the upper end of *a*, by good perforated corks. When the common air is expelled, the furnace is lighted. As soon as *a* is red hot, turn off the hydrogen by lifting away *g*, *i*, *k*, *l*, and plunge

into *f* the delivery tube of a similar apparatus fitted up for generating hydrochloric acid gas, and which must have been a short time in action, to expel common air. Hydrochloric acid gas is allowed to pass through *a* till the apparatus is quite cold, when the hydrogen is turned on again by lifting away the hydrochloric acid gas apparatus, and replacing the hydrogen apparatus. Hydrogen is passed through *a* till all traces of hydrochloric acid gas are expelled from the apparatus.

The flasks in which the hydrogen and hydrochloric acid gas are generated should be roomy, and supplied with a sufficient stock of material to keep up a brisk current of gas during the whole time of the operation.

After a short time the boat is taken out and weighed. It contains alumina alone. The iron may, of course, be calculated from the loss. It has been deposited in the lower part of the tube as a chloride. To extract it, attach to the upper end of the tube a retort filled with water acidulated with hydrochloric acid, and boil gently. The balloon at the lower end of the tube is previously turned, so that its tubulure may vent in a small porcelain capsule. The chloride of iron thus collected is mixed with a little sulphuric acid, evaporated to dryness, calcined in a platinum crucible, and weighed as peroxide. If manganese is present, it will be mixed as chloride with the chloride of iron. For their separation the two oxides are converted into sulphates by treatment with sulphuric acid, and a mixture of nitric and oxalic acids in atomic proportions. The mixture is evaporated in a balanced

platinum crucible, till the greater part of the free sulphuric acid is driven off. A few drops of nitric acid are added to peroxidize the iron, and the whole is moderately ignited. When no more loss of weight is perceived, the crucible is covered with its lid, and allowed to cool under a bell jar. It is now weighed; water is poured into the crucible, to dissolve out the sulphate of manganese. The oxide of iron is washed, reignited, and again weighed. This second weight represents the iron, and, on subtraction from the former, gives the sulphate of manganese.

The main amount of manganese (which remained insoluble when B was treated with nitric acid) is also treated in its capsule with sulphuric and oxalo-nitric acids, covering the capsule with a funnel, which is afterwards washed into the capsule. When the evolution of carbonic acid ceases, it is evaporated to expel free sulphuric acid, slightly heated, and weighed as sulphate of manganese. Its weight, joined to the portion separated from the iron, generally represents the total of manganese in the substance.

The sulphate of manganese should never be heated beyond redness, which temperature it resists, whilst any persulphate of iron present is decomposed.

We now take up the liquid, A, containing the alkaline earths and alkalies.

The washings having been added to the original solution, *lime* is determined in the usual manner. *Baryta* and *strontia*, if present, are thrown down along with the lime; the mixed oxalates converted into carbonates by ignition, and separated as above.

The filtrate, after removal of the lime, is evaporated down (§ 10, 6), and the *magnesia* determined by means of oxalic acid (§ 9).

As a trace of *manganese* may accompany the *magnesia*, the latter is dissolved in a hot concentrated solution of nitrate of ammonia, when any manganese present remains undissolved, and its weight is deducted from that of the *magnesia*.

The alkalies are determined in the solution and washings after removal of the *magnesia*.

Chrome, if present, will be found as chromic acid in A, will be thrown down as sesquioxide along with the *magnesia*, and will remain undissolved after treatment with nitrate of ammonia. Its separation from the *manganese* which may accompany it, will be explained below (I. § 55).—*St. Claire Deville*.

§ 24. SULPHUR IN IRON AND COPPER PYRITES, BLENDE, ETC.

a. Dry at a very gentle heat, reduce to a very fine powder, and weigh out 15 grains. Mix this thoroughly with 150 grains of nitre, and 90 grains of carbonate of soda, both perfectly pure, dry, and in impalpable powder. Place the mixture in a large platinum crucible provided with a well-fitting cover, and apply heat very gradually till the whole is fused to a thin liquid. Maintain it in this state for a few minutes. If any deflagration commences, put on the cover and reduce the heat. When the fusion is complete, take off the crucible and plunge its base into cold water. This will enable the saline mass to be easily detached. Place the fused mass in a beaker, cover it with a

funnel, and dissolve in very dilute hydrochloric acid. Wash out the crucible with the same liquid, and add the washings to the solution in the beaker. Finally, when all effervescence is over, take up the funnel and rinse it into the beaker by means of the washing bottle.

The sulphuric acid in the solution is now determined either in the usual manner (I. § 2), or volumetrically (II. § 3), and from its amount the sulphur is calculated.

b. Instead of nitre and carbonate of soda we may also employ oxide of mercury in large excess, mixed with sufficient carbonate of soda to prevent a too violent reaction ; or—

c. Heat the samples in a flask with very strong nitric acid (a dilute acid may cause the loss of some sulphur in the form of HS), and add afterwards a little hydrochloric acid. When all, or nearly all, the sulphur has been oxidized (which may require several hours' digestion), the solution is diluted with water and thrown upon a filter, which has been previously very carefully dried and weighed. The filter is well washed, and the washings added to the filtrate, the sulphuric acid in which is determined in the usual manner. The filter with its contents (silica, and a portion of unoxidized sulphur) is dried very carefully below 212° Fahr., and weighed. It is then burned in a platinum crucible, and the ashes are again weighed. The difference between these two weights, *minus* the weight of the filter previously ascertained, gives the amount of unoxidized sulphur, which is added to the

quantity found by calculation from the sulphuric acid in the liquid.

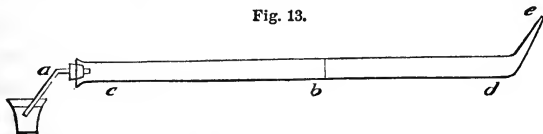
§ 25. METALLIC SULPHURETS—GENERAL METHOD.

a. Pulverize perfectly, and digest for some hours in a solution of potash heated to 122° – 140° Fahr. A current of chlorine gas is then passed into the solution. Sulphate of potash is formed, whilst the metal remains insoluble as peroxide or sesquioxide. The liquid is filtered off from the insoluble matter, and the sulphuric acid determined as usual. This method is applicable when lead is present; but zinc-blende renders the determination less accurate.

The amount of sulphur in commercial samples of that article, in organic substances, and in precipitates of the sulphurets of arsenic and antimony obtained in analysis, may also be determined in this manner.

b. Or (applicable also to organic sulphuretted bodies, liquids, &c.) take an ordinary combustion tube—fig. 13—put into it first from 30 to 45 grains of pure oxide

Fig. 13.



of mercury, then a little of a mixture of pure carbonate of soda and oxide of mercury in equal weights. The substance to be analyzed, if a solid, is carefully dried and very finely powdered, and 10 to 15 grains thereof are accurately weighed out, and thoroughly mixed with some of the above mixture of equal parts

carbonate of soda and oxide of mercury, so as to fill about half the tube. The front part of the tube is now loosely filled with the same mixture of soda and mercury to within about an inch of the open end. A plug of asbestos is introduced and the tube closed with a cork, through a hole in which passes the small bent tube *a*, dipping into water. The tube is laid in a horizontal position, and gently tapped and shaken longitudinally so that it may lie loosely.

The tube is now placed in the channel of Hoffmann's gas furnace, and the jets are kindled so as to heat about two inches of the tube at *b*, just in front of that part where lies the body to be analyzed. At the same time heat another portion about *c*, but less strongly than at *b*. As soon as *b* has arrived at a bright red heat, the heating is extended towards *d*, which it should reach in about fifteen minutes. At the same time the parts between *b* and *c* are gradually heated, and when the whole of the tube is at a bright red heat, the pure oxide of mercury at *e* is ignited. When this has quite disappeared, the tube is let cool, and its contents are emptied into a beaker and dissolved in water. If no oxide of mercury has escaped decomposition, a few drops of chloride of mercury are added. Add some muriatic acid to the fluid in the beaker, and wash the tube well out with it. If any sulphide of mercury is present, warm the solution, and pass the clear liquid through a filter; add some more muriatic acid to what remains in the beaker, together with a small crystal of chlorate of potash, and warm slightly again. If any trace of sulphide of mercury is left upon the filter, it

also is put into the liquid. Thus every trace of sulphide is perfectly oxidized in a short time. Filter the solution, and determine the sulphuric acid in the usual manner.

As it is almost impossible to obtain carbonate of soda absolutely free from sulphur, the following correction is required:—Make a mixture of perfectly dry, finely powdered carbonate of soda and oxide of mercury, equal parts, and grind them thoroughly together. Put about 600 grains of the mixture into a combustion tube as above, and determine its sulphur; and in all analyses made with this mixture make a proportionate deduction.

For volatile sulphuretted liquids, take a very thin glass tube, closed at one end, and accurately weighed: put into it some of the liquid in question, seal up the other end, and weigh again to find the quantity operated upon. Place it in the combustion tube near the closed end, and fill up with the mixture. Before commencing to heat, a thin glass rod is pushed up the combustion tube so as to come in contact with the bulb. Heat is applied in three different places; about the middle, towards the mouth, and at an intermediate spot, but most strongly to the first mentioned. As soon as the mercury has begun to volatilize from this part, the glass rod is pushed against the bulb, breaking the fine drawn-out point against the closed end of the combustion tube. The rod is withdrawn, and the cork with a bent tube inserted. Heat is now applied cautiously towards the bulb, so as to drive the liquid out of the bulb, but not immediately evaporate it. The

fire is now gradually advanced towards the end, and at the same time more and more of the front of the tube brought into a state of redness.

Note.—The bulbs used in this process are like small test-tubes, sealed at one end, and drawn out at the open end into a very fine capillary tube. To fill them, they are heated over a gas flame, and then plunged with the open point downwards into the liquid, which immediately rushes into the bulb. They are then sealed up before the blowpipe, and introduced into the combustion tube with the capillary point first.

Note.—The permanganate of potash cannot be used for oxidizing the sulphur in the more compact metallic sulphurets, pyrites, &c. However finely these may be powdered, and however long they are digested with large excess of the permanganate, a large part of the sulphur still escapes oxidation.

§ 26. MANGANESE.

Manganese, if alone, or only accompanied by the alkalies, may be determined by adding to the heated solution carbonate of soda. It is filtered, washed in hot water, dried, ignited, and weighed as brown mangano-manganic oxide, of which 100 parts represent 84 of metallic manganese. The ignition should be strong and rather protracted.

Ammoniacal salts if present in the solution should be destroyed by long boiling with carbonate of soda in excess.

§ 27. MANGANESE FROM IRON.

The solution, in which the iron must exist as peroxide, is freed by evaporation from any great excess of acid, and boiled with an excess of litharge. When cool, solution of sulphate of soda is added in excess, the whole is filtered, and the precipitate well washed. The filtrate is treated with a current of HS ; it is

then again filtered, boiled, and the manganese contained in it is determined by carbonate of soda as in I. § 26.

§ 28. ZINC.

a. Zinc existing alone, without any other fixed base or non-volatile acid may be determined by dissolving in pure dilute sulphuric acid, evaporating to dryness, and calcining at 752° Fahr. The residue is weighed as anhydrous sulphate of zinc, 80.2 parts of which represent 32.2 of metallic zinc.

b. If the zinc exists as, or can be converted into nitrate, it may be evaporated to dryness, ignited, and weighed as pure oxide of zinc.

c. To the solution carbonate of soda is added in excess, the whole boiled, the precipitate filtered, washed, &c. &c., and weighed, after strong ignition, as oxide.

If ammoniacal salts are present the carbonate of soda is added in very large excess, the whole boiled down to dryness in a flask placed in an inclined position. Plenty of hot water is poured on the dry residue, the whole once more boiled, and the carbonate of zinc filtered as above.

§ 29. ZINC FROM IRON.

If these are sulphates, evaporate to dryness, and calcine in a muffle to reduce to the state of oxides. Weigh them, dissolve in concentrated nitric acid, evaporate to dryness, and heat gently on the sand-bath as long as vapours of nitric acid are given off. Treat the mixture with nitrate of ammonia and a few drops

of caustic ammonia. The zinc is dissolved and decanted off. The residual oxide of iron is washed, dried, and weighed. The weight deducted from the joint weight of the oxides shows the amount of oxide of zinc; or—

Convert the iron into a per-salt by boiling with nitric acid; if the solution be too acid, add ammonia until it is but moderately acid. Then add acetate of soda in excess, and boil in a flask for an hour, until the solution is quite colourless. Filter; wash the reddish-brown precipitate with hot water. The whole of the zinc free from iron remains in solution, and is separated from soda as below, I. § 34.

§ 30. ZINC FROM MANGANESE.

Convert into nitrates (either by the direct action of the acid or by precipitating the mixed solution with carbonate of soda, with the due precautions—I. § 28—and dissolving the washed oxides in nitric acid), evaporate to dryness, and proceed as in I. § 29. Acetic acid may also be used to take up the zinc, leaving manganese untouched.

§ 31. ZINC FROM ALUMINA.

a. The neutral solution, or the yet moist mixed precipitate, is treated with cyanide of potassium in excess. Alumina is left undissolved as hydrate, which is filtered off.

b. Dissolve in nitric acid, evaporate to dryness, and heat to about 480° Fahr., till acid fumes are no longer given off. Nitrate of ammonia, with a few drops of

caustic ammonia at a gentle heat, extracts the zinc, which is decanted off. The residue of alumina is washed, dried, and weighed.

§ 32. ZINC FROM MAGNESIA, LIME, AND STRONTIA.

The mixed solution is precipitated with carbonate of soda in slight excess. Cyanide of potassium is then added in sufficient quantity, and the whole heated. The earthy carbonates remain insoluble, and are filtered off, while zinc dissolves. The filtrate is mixed with muriatic acid and boiled, gradually adding nitric acid till all hydrocyanic acid is expelled. The zinc is then determined with carbonate of soda (I. § 28), or as in I. § 34.

§ 33. ZINC FROM BAYRTA AND LEAD.

Sulphuric acid is added, and the solution concentrated. The soluble sulphate of zinc is removed by filtration, and determined by one of the methods given above.

§ 34. ZINC FROM ALKALIES AND REMAINING EARTHS.

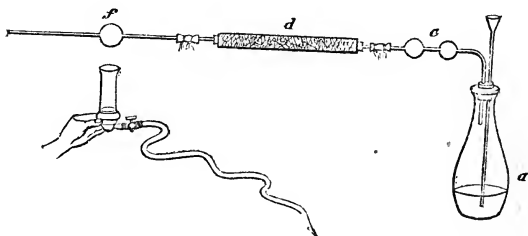
The solution (which is, if possible, freed from excess of acid by evaporation to dryness and resolution) is mixed with ammonia and hydrosulphate of ammonia. The precipitate is allowed full time to settle, the clear liquid is decanted off, the precipitate thrown upon a filter, washed with very dilute hydrosulphate of ammonia, dissolved in muriatic acid, digested until the odour of HS has disappeared, filtered, and the zinc in the filtrate determined as in I. § 28.

§ 35. COBALT.

The solution, which must contain no ammoniacal salts, is made hot, and precipitated with solution of pure potash; the liquid, which should not be too concentrated, is digested a little, and filtered. The precipitate is most carefully washed with boiling water, dried, ignited, and weighed as oxide, 37·5 grains of which represent 29·5 of metallic cobalt.

Where great accuracy is required, it is not sufficient to weigh the oxide, as on ignition it takes up variable quantities of oxygen. To find how much metallic cobalt is really contained in the ignited oxide, a quantity of it is introduced into the bulb *f*—fig. 14—

Fig. 14.



of a reduction tube, previously weighed. All loose particles not in the bulb being cleaned away with a feather, it is again weighed, and a current of hydrogen gas, generated from pure zinc and dilute sulphuric acid in a flask, *a*, and passed through a double bulb-tube, *c*, and a chloride of calcium tube, *d*, is allowed to traverse the reduction tube. As soon as the ap-

paratus is entirely freed from common air, heat is applied to the bulb, *f*, by a gas or spirit lamp, and continued as long as any watery vapour passes from the open extremity of the tube. The heat is gradually increased to full redness. When no more vapour passes over, any drops which may have condensed near the open end of the tube, are chased out by the flame of a lamp, and the apparatus is allowed to cool, hydrogen gas being still passed through. When quite cold, the reduction tube, with its contents, is again weighed. The loss shows the amount of oxygen in the portion of oxide acted upon, and, of course, the proportion of metallic cobalt in the whole.

The oxide may be put in a small tray of platinum or porcelain, which is then slid into the reduction tube, and again drawn out when the process is finished.

§ 36. COBALT FROM ZINC.

a. Add to the solution cyanide of potassium till the precipitate is redissolved. Then add muriatic acid in excess to form a white precipitate. Boil till this redissolves, and the liquid smells no longer of prussic acid. Add caustic potash; a precipitate forms and redissolves. Heat and pass a current of HS through the liquid. The zinc falls down, and the cobalt remains in solution; or—

b. The solution of the two metals (in nitric or muriatic) is largely diluted with water, and almost perfectly neutralized by carbonate of soda. For this purpose the latter is added in slight excess, and the precipitate which falls is redissolved in a few drops

of acid. A current of SH is then made to pass through the liquid, and a few drops of a dilute solution of acetate of soda added to complete the precipitation of the zinc. An excess of acetate must be avoided, and the liquid must not be heated. A fresh current of SH is now passed, until the precipitate does not seem to increase. The beaker is left to stand at a common temperature for twelve hours; then the precipitate is filtered and washed. To make sure that all the zinc has been thrown down, a drop of acetate of soda may be added to a little of the filtrate, which should then remain clear when SH is passed through. The cobalt is then thrown down from the filtrate by potash.

This method is applicable also to the separation of nickel from zinc.

If iron was present in the original solution (which in this case is preferably muriatic), it must be perfectly peroxidized by heating, and adding from time to time a little chlorate of potash. An excess of carbonate of baryta is then stirred into the liquid, without heat, and the whole allowed to stand. Peroxide of iron is thrown down along with the undissolved carbonate of baryta, and filtered off. The excess of baryta is removed from the filtrate by the cautious addition of sulphuric acid.

§ 37. COBALT FROM IRON.

Treat with muriate of ammonia and ammonia in large excess (care being taken that all the iron shall be peroxidized); filter off the precipitate of oxide of

iron, and wash well. Evaporate the filtrate to dryness, and heat to expel ammoniacal salts. Redissolve in a suitable acid, and determine the cobalt as above.

§ 38. COBALT FROM MANGANESE.

Convert into nitrates, evaporate to dryness, and heat as long as acid vapours are given off. Weigh the residue, and treat with dilute nitric acid. The cobalt dissolves, and is determined as above; the manganese (peroxide) remains undissolved, and is washed, dried, and weighed.

§ 39. COBALT FROM ALUMINA.

Treat with excess of ammonia, digest, and filter. The bulk of the cobalt remains in solution. As the precipitate of alumina is apt, however well washed, to retain traces of alumina, it is dried, calcined, and treated with hydrogen gas (I. § 35). The residue is treated with dilute nitric acid, which dissolves out the traces of cobalt, leaving the alumina pure.

§ 40. COBALT FROM MAGNESIA.

The solution is precipitated by a mixture of chloride of soda and caustic potash. The washed precipitate is digested whilst moist with solution of corrosive sublimate in excess, at a temperature of about 100° Fahr. The whole is then filtered, the filtrate and washings containing the magnesia are evaporated to dryness in a platinum capsule, and heated to expel the mercury. The oxide of cobalt is also heated to remove mercury, and determined as usual.—*Ulgren*.

§ 41. COBALT FROM LIME, BARYTA, AND STRONTIA.

The acidified solution is mixed with cyanide of potassium in excess. The whole is heated and mixed with carbonate of soda. Cobalt remains in solution, and is filtered off from the earthy carbonates.—*Haidlen.*

§ 42. COBALT FROM ALKALIES.

The solution, which should contain no ammoniacal salts, and should be neutral, is mixed with hydrosulphate of ammonia containing sulphur in excess, and the precipitate filtered off and washed with water containing a little hydrosulphate of ammonia. The bottom of the filter is then burst, and the sulphuret of cobalt washed into a beaker with muriatic acid. Nitric acid is then added, and the whole digested until the cobalt is perfectly dissolved. The filter paper is meantime dried, burnt to ash, and the ash put in the beaker. The solution is diluted with water, filtered, and the cobalt determined as usual in the clear liquor.

§ 43. NICKEL.

If alone, or only accompanied by alkaline or ammoniacal salts, evaporate the solution to dryness, powder finely along with a little more than its own weight of dry carbonate of soda, and ignite in a platinum crucible. The fused residue, carefully extracted with hot water, washed by decantation, dried, and ignited, yields pure oxide of nickel.—*Smith.* Or—

Heat the solution, add pure potash, digest a short

time, filter, wash very well with hot water, dry, ignite, and weigh as oxide, containing 78.6 of nickel; or—

Add to the solution hypochlorite of soda, and boil. Filter off the precipitate (peroxide of nickel), wash, dry, ignite to whiteness in Griffin's blast gas furnace, and weigh as oxide.

§ 44. NICKEL FROM COBALT

a. Dilute the solution very considerably with water, saturate it thoroughly with chlorine gas, and digest in the solution an excess of precipitated carbonate of baryta for about 24 hours. The whole of the nickel remains in solution, and is filtered off. The precipitate (sesquioxide of cobalt, and excess of carbonate of baryta) is redissolved in muriatic acid, and the baryta precipitated by sulphuric acid and filtered off.—*Rose*.

b. Pour the mixed solution into cyanide of potassium, boil, and precipitate the nickel from the solution by means of peroxide of mercury. Neutralize the filtrate with nitric acid, and add a neutral solution of pronitrate of mercury. The cobalt now falls as cobalticyanide of mercury, which is filtered, washed, dried, and ignited to remove mercury and decompose cyanogen.—*Liebig*.

§ 45. NICKEL FROM ZINC.

a. As in I. § 36.

b. Concentrate the solution, add potash in excess, and cyanide of potassium enough to redissolve the precipitate. Add mono-sulphide of potassium. Sulphuret of zinc is thrown down, and is filtered off and

washed. The filtrate is mixed with nitro-muriatic acid to destroy cyanides, and precipitated with potash.

§ 46. NICKEL FROM IRON. (See I. § 37.)

§ 47. NICKEL FROM MANGANESE. (See I. § 38.)

§ 48. NICKEL FROM ALUMINA. (See I. § 39.)

§ 49. NICKEL FROM MAGNESIA. (See I. § 40.)

§ 50. NICKEL FROM LIME, BARYTA, AND STRONTIA.
(See I. § 41.)

§ 51. NICKEL FROM ALKALIES.

The dried mixture is heated in a current of hydrogen gas. The alkaline salts are afterwards washed out from the reduced nickel, which is then weighed.

§ 52. CHROME.

From salts of chromic oxide, it is precipitated from a hot solution by ammonia, well washed, dried, ignited in a covered platinum crucible, and weighed as sesquioxide, containing 70 per cent. of chromium. Compounds of chromic acid are dissolved in muriatic acid, concentrated, and boiled, adding small successive portions of muriatic acid and alcohol till the chromic acid is entirely reduced. The alcohol is then expelled by heat, and ammonia added as above.

§ 53. CHROME FROM COBALT AND NICKEL.

Add to the mixed solution potash, and heat in a

flask till the supernatant liquid becomes colourless, and the chromic oxide is deposited. Filter, wash, and dry the mixed oxides, and apply a strong heat. Weigh the residue, pulverize, and submit a weighed portion of it to the action of nitric acid or muriatic acid. Cobalt and nickel dissolve, and are decanted off. The residue of chrome, which, if the heat has been high enough, is unaffected by the acid, is washed and weighed as sesquioxide.

If the oxides be in combination with any volatile acid, they may be heated at once without previous treatment with potash; or—

Precipitate with ammonia, filter off the clear liquid, which will contain nearly all the cobalt and nickel. Ignite the precipitate, and treat with nitric acid as above, adding the solution to the ammoniacal filtrate.

If the chrome be present as acid, it must first be reduced by muriatic acid and alcohol.

§ 54. CHROME FROM ZINC.

The separation is effected on the principles laid down in I. § 53. Ammonia dissolves the bulk of the zinc, and the rest is separated from the chrome after ignition by nitric acid.

§ 55. CHROME FROM IRON AND MANGANESE.

Precipitate the solution with ammonia, dry, weigh the filtered oxides, and ignite them in a current of dry hydrochloric acid gas. Manganese and iron are volatilized as chlorides, and condense in a cold part of the tube or in water, into which they may be led.

Chrome remains unchanged in the tray. A portion of the manganese will have escaped precipitation by the ammonia, and must be determined in the filtrate. (See also I. § 17).—*Deville*.

§ 56. CHROME FROM ALUMINA, EARTHS, AND ALKALIES.

Chrome is separated from alumina and magnesia as in I. § 23. From baryta, lime, and strontia, it may be separated by precipitation with ammonia, as also from the alkalies. Alkaline chromates are of course reduced in the ordinary manner.

§ 57. URANIUM.

Convert lower oxides into uranic oxide by heating with nitric acid. Add ammonia, filter, and wash with a dilute solution of sal-ammoniac. Ignite at a very high temperature, and cool quickly by placing the crucible on a block of cold metal. Weigh as black oxide— $U^4 O^5$.

§ 58. URANIUM FROM COBALT, NICKEL, MANGANESE,
AND ZINC.

To the mixed solution add bicarbonate of potash in excess; uranium remains in solution whilst the remaining oxides are thrown down. From the filtrate potash throw down the uranium, which is washed by decantation, then redissolved in muriatic acid, and precipitated with ammonia.—*Ebelmen*.

§ 59. URANIUM FROM IRON.

Precipitate both by an alkaline, wash the precipi-

tate, treat it with sulphurous acid, and boil. The iron remains in solution, whilst the uranium falls as a basic sulphite; 100 parts of which contain 70·89 of uranic oxide.

§ 60. URANIUM FROM ALUMINA.

The mixture is treated with excess of solution of potash. Alumina dissolves whilst uranium remains unattacked.

§ 61. URANIUM FROM MAGNESIA.

Muriate of ammonia is added to the neutral solution, and the uranium is precipitated with hydrosulphate of ammonia thoroughly saturated with sulphur.

§ 62. URANIUM FROM LIME, STRONTIA, AND BARYTA.

These earths are removed from uranium by means of sulphuric acid, alcohol being added in case of the two former.

§ 63. URANIUM FROM ALKALIES.

Precipitate with ammonia.

§ 64. VANADIUM.

The solution, whether of vanadic oxide or vanadic acid, is evaporated to dryness and ignited in a current of hydrogen gas. Suboxide of vanadium remains, 100 parts of which = 89·5 of metallic vanadium.

§ 65. VANADIUM FROM URANIUM, NICKEL, COBALT, ZINC, IRON, MANGANESE, CHROME, AND ALUMINA.

From these and all bases precipitable by, but not

soluble in, an excess of hydrosulphate of ammonia, vanadium is separated by means of this reagent well sulphured.

§ 66. VANADIUM FROM MAGNESIA.

Convert into nitrates.

§ 67. VANADIUM FROM LIME, STRONTIA, AND BARYTA.

Fuse with bisulphate of potash ; extract the fused mass with hot water, adding alcohol in case of the two former. Vanadate of potash is filtered off from the insoluble sulphates.

§ 68. VANADIUM FROM ALKALIES.

Dissolve in muriatic acid, and digest with sugar, till the vanadic acid is reduced ; then precipitate with ammonia, filter, wash with very dilute ammonia, and treat the dried vanadic oxide as above.

§ 69. TUNGSTEN.

Evaporate to dryness, ignite strongly, and weigh as tungstic acid.

Compounds *soluble* in acids are digested in fine powder in muriatic acid, till the insoluble residue (tungstic acid) assumes a yellow colour. It is then filtered off, washed, dried, and ignited. The solution is rendered ammoniacal, and treated with hydrosulphate of ammonia. The residue of the tungsten remains in solution, is evaporated to dryness, and ignited as above. In this manner tungsten is sepa-

rated from all bodies precipitable by hydrosulphate of ammonia, but insoluble in excess.

If the compounds are *insoluble* in acids, mix in fine powder with five parts bisulphate of potash, and bring it gradually to low redness.

From lime, strontia, and baryta it is separated by digestion in nitric acid, and subsequent treatment with carbonate of soda. The earthy carbonates are thrown down, whilst tungstic acid is estimated as loss.

From alkalis tungstic acid is separated by saturating with nitric acid, and decomposing with subnitrate of mercury. The precipitate is filtered off, washed, dried, ignited, and weighed as tungstic acid.—*Berzelius*.

From oxide of tin (with which it is often associated) tungsten may thus be separated. A known quantity is heated to redness in a porcelain crucible, whilst a current of dry hydrogen gas is passed in through an aperture in the cover. The calcined mass is boiled in muriatic acid, and sulphuret of tin thrown down by a current of HS; for the remaining treatment of which see below. The oxide of tungsten is washed, dried, and ignited.—*Rose*.

§ 70. TITANIUM.

Add to the solution ammonia, set the whole aside in a warm place to get rid of excess, filter, dry well, ignite, and weigh in a well-closed platinum crucible as soon as cool, as titanous acid.

From oxides precipitable by hydrosulphate of ammonia titanium is separated thus:—Add tartaric acid

and supersaturate with ammonia; no precipitate falls if sufficient tartaric acid has been added. Add hydrosulphate of ammonia, filter off the sulphurets, wash them with dilute hydrosulphate of ammonia, and convert them into oxides (I. § 42).

Titanium remains in solution, and if no other fixed body is present, it is evaporated to dryness and ignited, placing the crucible in an inclined position to burn off the carbon of the tartaric acid. If *lime* is present, the residue must be treated like an insoluble silicate—which see.

From *magnesia* titanium is separated by ammonia, muriate of ammonia having been added to retain the magnesia in solution. From lime and the alkalies it is likewise thrown down by ammonia. From *baryta* and *strontia* it is separated by sulphuric acid, in which it remains dissolved, and which, after filtration, is expelled by heat.

The remaining bases and metallic acids may, as a general rule, be separated from the foregoing by a current of HS passed through the acid solution, although in particular cases other means of separation may be preferable.

The sulphurets thus thrown down cannot, however, be weighed as such, as they either contain free sulphur or are mixtures of various stages of sulphurization. They are therefore generally redissolved in an acid, nitric, muriatic, or nitro-muriatic, and from the solution the oxide is thrown down by some other reagent.

§ 71. CADMIUM.

Add to the solution carbonate of soda, filter, wash, dry, ignite, and weigh as *oxide* of cadmium. The filter must be burnt separately, as the oxide is very easily reduced.

§ 72. BISMUTH.

The solution is precipitated with carbonate of ammonia in excess, and set to digest on the sand-bath. It is then filtered, washed, dried, and ignited—the filter being burnt separately, and weighed as oxide. The solution must contain no muriatic acid.

From cadmium bismuth is separated by ammonia, which dissolves the former oxide, leaving the latter untouched.

When bismuth is to be separated by HS from oxides not precipitable thereby, the solution when diluted should be mixed with acetic acid.

§ 73. LEAD.

a. To the solution is added oxalate of ammonia. The liquor should be neutral and free from ammoniacal salts. Filter, &c., and ignite in a porcelain crucible, burning the filter separate. Weigh as protoxide.

Carbonate of ammonia, mixed with a little caustic ammonia, may be used instead of the oxalate.

b. Add to the solution dilute sulphuric acid in excess, wash with dilute alcohol or exceedingly dilute sulphuric acid, and heat in a platinum crucible to expel all free acid. Weigh as sulphate of lead.

From *bismuth* lead may be separated by passing a current of HS through the acid solution at a rather high temperature. Sulphuret of lead dissolves in the acid, whilst that of bismuth is precipitated and filtered off; or—

The two are precipitated as carbonates by carbonate of ammonia, dissolved in acetic acid, and placed in a flask. A piece of pure lead of known weight is introduced, submerged entirely in the liquid, the flask is corked and set aside for some hours. When the whole of the bismuth is precipitated in the metallic state, the lead is withdrawn, washed, and weighed. The bismuth is collected in a filter, washed with water which has been boiled, and cooled in a stoppered vessel, dissolved in nitric acid, and determined as usual. The lead solution is precipitated with carbonate of ammonia as above. From the oxide of lead a quantity corresponding to the loss sustained by the metallic lead is subtracted.—*Ulgren*.

From *cadmium* lead is separated by treating the solution with cyanide of potassium in excess and applying heat. Lead separates, whilst cadmium remains in solution. The former is redissolved in nitric acid and determined as usual. The liquid is boiled with muriatic acid, and the cadmium then determined as usual.

When lead has been thrown down by HS from a solution, the washed precipitate is dissolved in strong muriatic acid as long as fumes of HS are given off. Nitric acid is next added; the whole evaporated to dryness, put in a porcelain crucible, sulphuric acid is

added, and heat is applied till the excess of acid is expelled. The sulphate of lead is then weighed.

§ 74. COPPER.

To the solution, which should be hot and moderately dilute, an excess of caustic potash is added and the whole boiled, preferably in a platinum capsule. The brownish-black precipitate is filtered, washed with boiling water, dried, ignited, and weighed as protoxide.

If any portions adhere to the side of the capsule after long boiling, they are dissolved off with dilute sulphuric acid, diluted with water, and boiled afresh with potash, and added to the rest in the filter.

If ammonia is present, part of the precipitate is liable to redissolve on long standing.

From *lead* copper is separated by sulphuric acid, the oxides being dissolved in nitric acid, mixed, evaporated to dryness, heated to expel acid, and the dry mass treated with water, which extracts the sulphate of copper. The insoluble lead-salt is filtered off, &c., and weighed. In the filtrate the copper is determined as usual; but, as a little lead sometimes remains in solution, the liquid, after removal of the copper, is neutralized with an acid, and mixed with oxalate of ammonia. The trace of lead thus thrown down is added to the rest; or—

The sulphurets being thrown down together by HS, are treated with cyanide of potassium. Sulphuret of copper dissolves, whilst sulphuret of lead is

untouched. The solution of copper is then decomposed by nitro-muriatic acid, and precipitated by HS.

From *bismuth* copper is separated by carbonate of ammonia, in which the latter oxide dissolves alone. The whole is allowed to digest for some time, and the filtered precipitate is washed with carbonate of ammonia.

From *cadmium* copper is separated as from bismuth. Or: The solution of the two oxides in excess of cyanide of potassium is treated with HS, which throws down the copper alone.—*Haidlen*.

From the other oxides above mentioned copper is separated by passing HS through an acid solution until perfectly saturated with the gas. The sulphuret is rapidly filtered, washed uninterruptedly with HS water, dried, detached from the filter, put into a beaker, and left to digest in nitro-muriatic acid. The filter is burnt and its ashes added.

When the decomposition is complete, the liquid is diluted with water, filtered from the sulphur, and determined with potash as above.

The sulphuret of copper, instead of being thus decomposed, may be heated in a glass or porcelain tube in a current of hydrogen. It is thus reduced to subsulphuret, Cu S^2 , which contains exactly the same per centage of copper as an equal weight of the oxide, 40 parts containing 32 of metallic copper.—*Brunner*.

In some cases copper is imperfectly separated from other metals, especially zinc and nickel, by a current of HS. The following methods are therefore recommended:—

§ 75. COPPER FROM ZINC AND NICKEL.

Acidulate the mixed solution with sulphuric acid, heat it to boiling, and add hyposulphite of soda. Sulphuret of copper is thrown down free from zinc. The other oxides remain in solution.

§ 76. COPPER FROM MANGANESE, IRON, ZINC, NICKEL, COBALT, ARSENIC, AND ANTIMONY.

Pass through the acid solution sulphurous acid gas in excess, and add hydriodic acid. The copper falls as white diiodide, which may be decomposed with nitric acid and treated in the usual way. Bismuth, if present, will fall down along with the copper.

§ 77. MERCURY.

Dissolve in nitro-muriatic acid, and add pure potash till but a very slight acid reaction remains. Add formiate of soda, and let the whole stand for some days at a temperature not exceeding 160° Fahr. The mercury is precipitated as subchloride (calomel), and is thrown upon a balanced filter and washed. More formiate of soda is then added to the filtrate, which is set aside for another day, and any further precipitate which may form is added to the former. The whole is then dried at a gentle heat till its weight remain constant. 100 parts represent 84.96 of metallic mercury.

In many amalgams mercury may be estimated as loss by applying heat sufficient for its volatilization.

From *bismuth* and *cadmium* mercury is separated by

applying an excess of cyanide of potassium, and afterwards dilute nitric acid, and boiling the whole. The excess of hydrocyanic acid is expelled, and carbonate of potash now throws down the bismuth and cadmium. The mercury remains in solution as cyanide, and is thrown down by HS.—*Haidlen*.

From *copper* mercury is separated by neutralizing the solution with carbonate of soda, and adding an excess of cyanide of potassium. From the solution the mercury alone is precipitated by hydrosulphate of ammonia. The precipitate is washed, placed in a moderate-sized flask provided with a stopper, covered with muriatic acid, and treated with a slow current of chlorine gas. As soon as the sulphur separates in yellow flakes the flask is gently heated to expel free chlorine, cooled, filtered, the precipitate (sulphur) washed, and in the filtrate the mercury determined by a formiate.

From *lead* mercury is separated by sulphuric acid, using a considerable excess, and heating the residue with dilute sulphuric acid before washing. From the other metallic oxides before mentioned, the alkalies and earths, mercury is separated by passing a current of HS through the acid solution, and treating the sulphuret thrown down as directed in the separation of mercury and copper.

§ 78. SILVER.

The solution, rendered acid by nitric acid, and not too concentrated, is mixed with muriatic acid in excess. It is heated. set aside for some hours, the clear liquor

decanted off and replaced by hot water. It is then filtered, using a small filter, washed with dilute nitric acid, and afterwards with pure water, dried, and ignited in a porcelain crucible. The filter, scraped as clean as possible, is burnt upon a crucible lid, the ashes moistened with nitric and muriatic acid, dried, heated, and put into the crucible. 143.4 of the precipitate represent 108 of silver.

If ammoniacal salts, or alkaline chlorides in general, are present, the filtrate should be evaporated nearly to dryness, treated with an excess of nitric acid, and heated again. If any chloride of silver has been taken up it will be deposited, and may be added to the main bulk.

Chloride of silver may be weighed upon a balanced filter, after having been very carefully dried at 212° Fahr., as long as any change of weight is observed.

Or the precipitate may be washed by decantation, and then introduced into a Rham's specific gravity bottle, which is then filled up with water and weighed. The difference between the weight thus obtained and that of the bottle when filled with distilled water is the amount of chloride of silver.—*Méne.*

Silver may be separated by muriatic acid from all oxides hitherto mentioned. If lead is present the solution must be largely diluted with water. Substances containing mercury must be brought to the state of per-salts with nitric acid.

§ 79. GOLD.

The solution should be treated with excess of mu-

riatic acid, and concentrated to expel nitric acid. Oxalic acid is then added, and the whole digested at a gentle heat for one or two days. The precipitate of metallic gold is filtered, washed, gently ignited, and weighed.

Instead of oxalic acid the reduction may be effected by protochloride of iron.

In either case the liquid should contain a sufficiency of muriatic acid.

Oxalic acid serves to separate gold from all the above-mentioned metals and oxides, except silver and lead.

From lead gold is separated by saturating the concentrated solution with ammonia and adding hydrosulphate of ammonia, well saturated with sulphur, in large excess. The whole is left to digest for some time in a covered vessel. The precipitate is then filtered off and washed, and the gold thrown down from the filtrate by supersaturation with dilute hydrochloric acid.

From *silver* gold is separated in different manners, according to the proportions. Where the silver is *below 15 per cent.*, the alloy, beaten out into thin leaf, is treated with nitro-muriatic acid diluted with water, heated, allowed to settle, the precipitate of chloride of silver filtered off, and the gold determined in the filtrate by oxalic acid.

If the silver is *above 80 per cent.*, dissolve in pure nitric acid with the aid of heat. Dilute and filter off the solution of silver. Wash, dry, heat, and weigh the residue of gold. Then dissolve it in nitro-muriatic

acid, dilute and filter off any trace of chloride of silver. If the proportions are intermediate the alloy is fused in a porcelain crucible with thrice its weight of pure lead. Proceed as in the last case, and finally determine the gold by means of protochloride of iron.

§ 80. RHODIUM.

Add to the solution carbonate of soda in excess, evaporate to dryness in a platinum capsule, ignite the dry residue, treat with water, filter the peroxide of rhodium, wash with muriatic acid and then with water, ignite and reduce by a gentle heat in a current of hydrogen gas.

When rhodium is thrown down from a solution by HS , and the precipitate of sulphuret filtered off, a second portion is generally obtained on heating and concentrating the filtrate.

§ 81. PALLADIUM.

To the solution is added a solution of cyanide of mercury. After some time a yellowish-grey precipitate forms, which is filtered, &c., ignited, and weighed as metallic palladium. If alcohol exist in the solution no other metal should be present.

From iron and all metals not thrown down by HS , palladium is separated by means of this reagent.

The sulphuret of palladium is roasted, dissolved in muriatic acid, neutralized with carbonate of soda, and determined as above.

From all metals whose sulphurets are insoluble in excess of hydrosulphate of ammonia, except silver and

copper, palladium may be separated by adding cyanide of potassium in excess, and passing a current of HS into the solution. Sulphurets of cadmium, bismuth, &c., are precipitated, whilst the palladium remains in solution.

From *copper* palladium is separated by diluting the nitric solution with water and adding formiate of alkali, and boiling as long as gas is given off. Metallic palladium is deposited and filtered off; copper remains in solution.

From *silver* palladium is separated by muriatic acid.

From the alkalies palladium is separated by igniting the mixed chlorides in the air, and separating the reduced palladium by washing.

§ 82. IRIIDIUM.

The concentrated solution is mixed with a strong solution of sal-ammoniac, and with a large quantity of alcohol. The precipitate is washed with dilute alcohol and ignited, when metallic iridium remains.

The separation of iridium from other metals turns upon the circumstance that it is precipitable by HS from acid solutions; its sulphuret being soluble in cold nitric acid and in hydrosulphate of ammonia.

§ 83. OSMIUM.

Osmium is precipitated from its solutions by formic acid.—(See “Platinum Ores” in Part II.)

§ 84. PLATINUM.

The solution is precipitated with an alkaline formiate, and the dried and ignited precipitate weighed as metallic platinum.

From *gold* platinum is separated by the process directed for the determination of iridium; the double chloride of platinum and ammonium is filtered off, ignited, and the residue of platinum weighed. The gold is determined in the filtrate by means of oxalic acid.

From *silver* platinum is separated by boiling in sulphuric acid. The silver dissolves; the platinum remains untouched.

From *lead* platinum is separated by treating the solution with sulphuric acid, by which the lead is precipitated.

From all metals whose chlorides are soluble in weak alcohol, *e.g.*, copper, iron, &c., platinum is separated as from gold.—(See also "Platinum Ores," Part II.)

§ 85. TIN.

Tin is determined by concentrating the solution, adding nitric acid in such excess as to expel any muriatic acid present, and boiling, when the sudden disengagement of red fumes marks the formation of insoluble peroxide, 10 parts of which contain 7.86 of tin.

Volumetric methods of determining tin will be found in Part II.

From all metals soluble in nitric acid, tin may be separated by dissolving and boiling, as already stated,

and filtering the oxide of tin from the soluble nitrates. If bismuth is present the wash water is mixed with nitric acid.

If gold or platinum, and its associates, be present, dissolve in nitro-muriatic acid, and heat in a current of dry chlorine gas. The chloride of tin volatilizes, and may be led into a flask of water by having the extremity of the tube beyond the flame bent downwards very close to the heated portion. Gold and platinum remain behind in the porcelain boat.

Those oxides not precipitated by HS are separated from tin by a long-continued current of that reagent. The sulphuret of tin thrown down is filtered, washed, partially dried, put in an uncovered crucible, and heated gently as long as sulphurous acid is given off. The heat is then increased to redness to convert the whole into peroxide.

As the sulphurets of tin are soluble in excess of hydrosulphate of ammonia, they may thus be separated from sulphurets of lead, copper, bismuth, cadmium, and mercury.

§ 86. ANTIMONY.

The solution in muriatic or nitro-muriatic acid is diluted with water, tartaric acid having been previously added to prevent turbidity. A current of HS is then passed through the liquid to saturation; it is let stand in a warm place till all smell of the gas has ceased, and the precipitate is then filtered, washed, and dried upon a previously weighed filter, and very carefully weighed.

A part of the dry sulphuret thus obtained is introduced into the bulb of a previously weighed reduction-tube. Any portion not lying in the bulb is removed with a feather, and the whole is carefully weighed to know the quantity of sulphuret operated upon. A current of hydrogen gas is passed through, and heat applied as in the reduction of cobalt, *q. v.* The sulphur deposited at the outer end of the tube is chased away with the flame of a flexible gas tube or small spirit lamp. When no more sulphur is deposited, and no more HS given off, the apparatus is cooled, disconnected, and the reduction-tube weighed. The loss of weight represents the sulphur present in the sulphuret of antimony operated upon. The heat should be very gentle at first, and be only raised towards the end for a short time.

Or a known weight of the precipitate (sulphuret of antimony) is treated with bisulphuret of carbon, which dissolves out free sulphur; it is then mixed with fifty times its weight of pure peroxide of mercury, and slowly heated in a porcelain crucible, moderating the temperature when grey mercurial fumes appear. When these have ceased, the temperature is again raised, and maintained until no further diminution of weight is remarked.

Antimoniate of antimony, Sb^2O^3 , remains as a white powder.—*Bunsen.*

From *tin* antimony is separated by dissolving in muriatic acid, boiling for a few minutes, and adding a saturated and boiling solution of chlorate of potash, till the whole is perfectly dissolved. A sheet of

zinc (pure) is introduced, which precipitates both metals in a state of fine division. The zinc is withdrawn, and washed into the liquid. Muriatic acid is now added, which dissolves the tin, leaving the antimony untouched. Collect and wash the latter.—*Levol*.

HS is used to separate antimony from all metals whose acid solutions it does not precipitate.

Those whose sulphurets are insoluble in excess of hydrosulphate of ammonia are separated thus:—The solution in muriatic acid, or, if necessary, in nitromuriatic acid, is diluted with water, and a current of HS is passed through. The washed precipitate is digested with excess of hydrosulphate in a corked flask. The liquid is then filtered, and the precipitate washed with very dilute hydrosulphate. The sulphuret of antimony is found in the filtrate.

From *gold* and *platinum* antimony is separated as follows:—A quantity of the alloy pulverized, or

Fig. 15.



reduced to the smallest possible fragments, is introduced into the bulb of a reduction tube, *a*—fig. 15—previously weighed. By a second weighing the amount of matter operated upon is determined. One limb of the tube is bent down at right angles, and dips into a bottle containing a solution of tartaric mixed with muriatic acid. A tube to allow the escape of gas from the bottle merely passes through the cork without dipping into the liquid.

Dry chlorine is introduced at the other end of the reduction tube, and when the air is expelled from the apparatus, heat is applied very cautiously to the bulb. When no more volatile matter passes over, the apparatus is allowed to cool. To prevent the liquor in the bottle from ascending into the bulb, which easily happens when the alloy has become red hot, and chlorine is eagerly absorbed—it is well to allow the end of the tube only just to touch the surface of the liquid, and to have a bulb blown on the descending limb of the tube. The source of heat should be withdrawn as soon as any part of the alloy is ignited. When all is over, the descending limb of the tube is cut off, and washed into the bottle, which contains all the antimony. The gold and platinum remain in the tube.

§ 87. MOLYBDENUM.

Molybdic acid, when contained in an alkaline solution, is neutralized with nitric acid, and precipitated with a solution of protonitrate of mercury. The liquid, after saturation with nitric acid, is allowed, if carbonates were present, to stand a day in a warm place, that the carbonic acid may escape. The precipitate is allowed to stand for some hours, collected on a weighed filter, washed with protonitrate of mercury, dried at 212° Fahr., and accurately weighed. The precipitate is then removed from the filter, and is heated in a platinum crucible, through an opening in whose lid a current of hydrogen gas is introduced. Too high a temperature should be avoided. The

residue is weighed as molybdic acid, 10 parts of which contain 7.5 of molybdenum.

HS throws down molybdenum very slowly from its solutions as sulphuret, which is soluble in excess of hydrosulphate of ammonia.

§ 88. ARSENIC.

Solutions of arsenic acid, containing no other body except nitric acid, are mixed with a weighed amount of recently-ignited protoxide of lead, evaporated to dryness, ignited in a weighed platinum crucible, and weighed. The excess of weight over that of the oxide of lead employed, shows the amount of arsenic acid. If arsenious acid be present, nitric acid is added along with the protoxide of lead, arseniate of lead being produced on ignition.

If substances are present which render this method impracticable, HS is passed through the diluted liquid to complete saturation. The beaker is set aside for excess of gas to evaporate. The whole is poured upon a filter, any traces of sulphuret of arsenic which may adhere to the glass having been dissolved off with a little carbonate of soda, and added to the rest. The sulphuret of arsenic is now washed, dried, and weighed, the filter having been previously balanced.

The bulk of the precipitate is now put into a flask, the filter being weighed again afterwards to know what quantity has been acted upon. A solution of pure potash is added, in which the sulphuret dissolves; afterwards a concentrated solution of permanganate of potash is added as long as it is decolorized, the flask

being heated all the time. When the liquid retains its purple colour it is acidified with muriatic acid, and the sulphuric acid determined after filtration. The amount of sulphur present in the original precipitate is calculated from the quantity of sulphuric acid thus found, and this amount being subtracted from the entire weight of the same precipitate, shows the quantity of arsenic present.

Instead of permanganate of potash, the precipitate may be digested in nitro-muriatic acid for a long time. The undissolved sulphur, after it has separated in lumps, is filtered off from the diluted solution, dried very carefully, and weighed on the balanced filter. The amount of sulphuric acid is then determined, and its corresponding amount of sulphur is added to the quantity already weighed.

It is not easy to precipitate the whole of the arsenic from a solution by means of HS. The filtrate should be again treated with the gas, and left to stand for some time, to see if a fresh portion may be deposited.

Arsenic acid may also be determined by taking a known weight of pure metallic iron (in excess) dissolving in nitric acid with the aid of heat, mixing with the arsenical liquor, and precipitating with ammonia in excess. The precipitate is filtered, washed, dried, ignited (very gently at first), and weighed. The amount of iron employed being known, its proportion of peroxide is subtracted from the weight of the ignited precipitate. The remainder is arsenic acid.—*Berthier*.

From oxides not thrown down by HS, arsenic is

separated by this means, the dilute solution being acidified by muriatic acid.

Arsenical alloys are in general best dissolved in nitro-muriatic acid.

The sulphurets of arsenic being soluble in excess of hydrosulphate of ammonia, this reaction serves to separate arsenic from all metals whose sulphurets do not thus dissolve. To the filtrate, which should be copiously diluted with water, muriatic acid is cautiously added, until the whole has a slightly acid reaction. The sulphuret of arsenic is thus precipitated.

Arseniates of lead, strontia, lime, and lead, may be determined by means of sulphuric acid and alcohol. The arsenic acid is determined as loss.

From *antimony* arsenic may be separated as follows:—The solution, if it has not been made with nitro-muriatic acid, is peroxidized by chlorine, or an alkaline hypochlorite. A large excess of tartaric acid is added, then sulphate of magnesia, and finally ammonia. The arsenic is thrown down as double arseniate of ammonia and magnesia; the antimony remains in solution.

The arsenical precipitate is dissolved in nitric acid, evaporated to dryness in a platinum crucible, and a known weight of pure magnesia added. The whole is agitated with a very little water, so as to form a thick mass; it is again evaporated to dryness, calcined, and weighed. The magnesia added having been deducted, 100 parts of the rest represent 48.01 of metallic arsenic.

From *gold* and *platinum* arsenic may be separated in the same manner as antimony.—I. § 86.

From *antimony* and *tin* arsenic is separated thus:—Oxidize with very strong nitric acid, evaporate the residue to dryness, and fuse in a silver crucible with nine times its weight of soda. Treat the fused mass with a little water, and add alcohol of specific gravity 0·83, till it forms one-third of the liquid. Set aside for twenty-four hours, stirring occasionally. Collect the precipitate on a filter, rinse the glass with dilute alcohol, and wash the precipitate first with equal volumes of water and alcohol of 0·83, then with one volume of water to three of alcohol, till the washings no longer give a precipitate, if acidulated with dilute sulphuric acid, and mixed with HS. The residue, antimoniate of soda, is dissolved in a mixture of muriatic and tartaric acids, and the antimony thrown down as sulphuret by HS.

Arsenic and tin remain in the filtrate, which is slightly acidified with muriatic acid, and thoroughly saturated with HS, set aside as long as anything is deposited, and then filtered. The filtrate is gently heated to expel free HS and alcohol, and is mixed with sulphurous acid and aqueous HS, when a second precipitate is often formed, which is added to the former.

The mixed precipitate (sulphurets of tin and arsenic) is now carefully dried, weighed (the filter having been previously counterpoised), and a known portion of it placed in a porcelain tray, inserted in a tube, and heated in a current of dry HS. The sulphuret of

arsenic is volatilized; the tin remains behind, and is dissolved, and estimated in the usual manner. The arsenic may either be estimated as loss on the weight of the original substance, or to the outer limb of the reduction-tube may be attached a Wills' bulb-tube, containing a solution of potash, into which any portion of the sulphuret of arsenic that may be deposited in the reduction-tube is driven by the application of heat. From this solution it may be again afterwards thrown down, by cautiously acidulating with muriatic acid.

Antimony and tin may also be separated from arsenic by dissolving the mixed sulphurets in sulphuret of potassium, precipitating this solution with a large excess of bisulphite of potash, digesting for some time in the water bath, and then boiling till all the sulphurous acid and two-thirds of the water are driven off. The precipitate left contains all the antimony, or tin, whilst the arsenic is found in the filtrate, and may be thrown down by HS.—*Bunsen*.

This method does not serve for the separation of tin and antimony.

§ 89. TELLURIUM.

Tellurous acid in solutions is mixed with a slight excess of muriatic acid if the liquor be alkaline. Nitric acid present must be expelled by concentration with the addition of muriatic acid in excess. If free sulphuric acid be present, it is evaporated to dryness, and fused in a platinum crucible. The acid liquid is now concentrated, heated in a flask not quite to boil-

ing, and a little sulphite of soda gradually added. The flask is corked and set aside for some days in a warm place. The clear liquid is poured upon a balanced filter, the black precipitate of metallic tellurium is washed in the flask with sulphurous acid water, which is also poured upon the filter, into which the precipitate itself is lastly poured, washed, dried at a gentle heat, and weighed.

Telluric acid may be reduced to tellurous acid by prolonged digestion in hot muriatic acid. Or, if no chlorides are present, the solution of tellurates may be mixed with nitrate of silver in slight excess, the precipitate redissolved in ammonia, the excess of which is removed by evaporation. Basic tellurate of silver falls down, is thrown upon a balanced filter, washed carefully, dried, and weighed. This precipitate contains 20·2 per cent. of telluric acid.

Tellurium being thrown down by HS from its solutions, and the precipitate being soluble in excess of hydrosulphate of ammonia, is thus readily separated from the majority of metals, the earths, and alkalies. The precipitate of sulphuret of tellurium is decomposed by nitro-muriatic acid, filtered off from free sulphur, and determined as above. From *gold* and *platinum* tellurium is separated by heating the mixture in a tube through which a stream of dry chlorine gas is passed, the vapours of chloride of tellurium being conducted into a flask containing water and some muriatic acid. From arsenic, antimony, and tin it is separated by precipitation with sulphite of soda.

§ 90. TANTALUM.

The compounds of tantalic acid are very finely powdered, dried, mixed with eight times their weight of bisulphate of potash, and fused in a platinum crucible till the whole forms a perfectly thin liquid, without any sediment. The mass is boiled in water as long as anything is taken up. Tantallic acid remains undissolved, and is filtered off and weighed. Traces of tin, tungstic acid, and iron, however, often remain with the tantalic acid. The two former are separated by prolonged digestion in hydrosulphate of ammonia, which dissolves them as sulphurets. Muriatic acid poured upon the washed residue extracts the iron.

From titanic acid tantalum is separated by mixing the powdered mass with sugar, carbonizing by heat, and treating with a stream of dry chlorine gas in a porcelain tube heated to redness. Both pass over as chlorides, and are received in a flask containing water, from which sulphuric acid precipitates the tantalic acid, leaving titanic acid dissolved.

§ 91. SILICA.

Solutions containing silica are acidified with muriatic acid, and evaporated to perfect dryness. The residue is extracted with water containing a little muriatic acid, which leaves the silica untouched. It is filtered off, well dried, ignited, and weighed.

Silicates capable of being attacked by acids are reduced to an impalpable powder, and digested with strong muriatic acid, at about 212° Fahr., in a beaker

covered with a glass plate. When no gritty particles are felt on drawing a glass rod over the bottom of the beaker, water is added, the whole evaporated to dryness, the residue extracted with water, and a little muriatic acid, filtered, washed, dried, ignited in a closed crucible, cooled over sulphuric acid in the drying apparatus, and weighed. If lead or silver be present, nitric acid must be employed instead of muriatic.

The water contained in many silicates should be determined in a separate portion.

§ 92. SILICATES—DEVILLE'S METHOD.

a. Silicates soluble in acids.

Those silicates only which form a jelly with acids should be considered soluble.

Place a portion in a platinum boat, heat it in a stream of hydrogen, and note the loss of weight. The iron is reduced to the metallic state, and sesquioxide of manganese to protoxide. Some silicates, such as epidotes, unattackable before this process, may now be reduced to a jelly by nitric acid.

In all cases silicates may be freed from their iron and manganese by prolonged ignition in a current of hydrochloric acid gas. These two metals are volatilized as chlorides, and may be received in a flask containing water. This is the most expeditious way of determining the iron and manganese in silicates, and shows at the same time the state of oxidation in which they exist. The residue is very often found attackable by acids. The general process is—

1. Put the weighed sample in powder into a flat-bottomed platinum capsule which can be covered with a flat plate of platinum, both accurately balanced; moisten with water and put it to digest in pure nitric acid, agitating frequently to prevent the powder from clotting.

2. Evaporate to dryness, and heat as in I. § 23, until the disengagement of acid vapours ceases. If manganese is present, heat more slowly, and remove when the black tint is uniform.

3. Moisten with nitrate of ammonia exactly as in § 23.

4. Decant the liquid containing the alkalies. When necessary a small filter is employed, which is only required to prevent any portion from being spilt. In this case, after washing, what has been stopped by the filter is replaced in the capsule. If, after being dried, it contain nothing, it is thrown away. The liquor containing the alkalies and magnesia may be received into the bottom of a flask cut off at the neck, and provided with a lip by heating the edge of the vessel at a glass-blower's lamp, and placing a triangular plate on the softened glass. The opening may be covered with a plate of platinum. The oxides and silica are washed with the precautions already described. The analysis now is divided into two branches.

5. The silica and oxides remaining in the capsule are treated with nitric acid, which removes the oxides, except the peroxide of manganese which should colour the silica a pure black.

6. The nitrates of alumina and iron are evaporated, and treated as in I. § 14, *a*; or I. § 23.

7. The manganese is dissolved away from the silica by sulphuric acid, the liquid decanted off, evaporated to dryness and weighed.

8. The silica left pure in the capsule is ignited and weighed.

9. The solution containing the magnesia and alkalies may be treated as in I. § 23.

After removal of the potash, lithia may be separated from soda by adding phosphate of soda, which throws down double phosphate of soda and lithia. This salt contains when calcined 12.32 per cent. of lithia. The soda is then estimated as loss on the original joint weight of the three alkalies; or—

The salts of lithium and sodium being converted

into chlorides, the weighed mass is digested in a mixture of alcohol and ether, which takes up the lithium.

The excess of platinum left after determining the potash may be removed by evaporating to dryness, igniting, and dissolving out the alkaline salts from the residue of metallic platinum.

b. Silicates insoluble in acids.

Reduce the silicate to coarse powder in an iron mortar, pass it through a silk sieve, digest in very dilute nitric acid (to remove iron taken up from the mortar), wash, and dry. It is now placed in a small platinum crucible, pure powdered carbonate of lime is placed upon it; the whole is heated to 392° Fahr. and weighed. The quantity of silicate introduced into the crucible should, of course, be very accurately weighed, as also the carbonate of lime introduced. The amount of carbonate of lime required varies from 30 to 100 per cent. according to the nature of the silicate operated upon. The powdered silicate and the lime are now carefully mixed together with a platinum spatula, which, together with the sides of the crucible, are cleaned with a crow's feather. Heat is now applied, at first sufficient to expel carbonic acid from the lime, and afterwards to fuse the whole to a perfectly limpid glass. The weight should be the same as when weighed after the heating to 392° Fahr., *minus* the carbonic acid expelled from the lime. There is no fear of loss by spirting or effervescence. The mass is then extracted by bending the crucible a little between the fingers, and is broken to fragments in an agate mortar covered with a fine cloth. It is now

decomposed by nitric acid, and the remainder of the process is as in the case of the soluble silicates. Iron and manganese, if present, should be removed by ignition in a current of hydrochloric acid gas *before* the treatment with lime. Chrome, if present, may be first ignited in a current of carburetted hydrogen, and afterwards in one of hydrochloric acid gas. Chloride of chromium is volatilized; or—

c. The finely powdered silicate is mixed with 1 part of pure fluor spar, and 4 to 5 parts of pure carbonate of lime, and heated in a platinum crucible capable of holding thrice the bulk.—*L. Smith.*

Substances of other kinds, unattacked by acids, are treated in the same manner as the insoluble silicates. Sometimes an alkaline carbonate is found more convenient than lime.

§ 93. SILICA FROM TIN, VANADIUM, TITANIUM, TANTALUM,
AND PHOSPHORUS.

From *tin* silica is separated by igniting a weighed quantity in a stream of hydrogen gas. The temperature should not exceed a dark-red heat. The loss of weight = the oxygen in the oxide of tin. The metallic tin is now dissolved out, either in a stream of chlorine gas, heat being again applied, or the mass is taken from the tube and digested in nitro-muriatic acid. Silica remains untouched.—*Rivot.*

From *vanadic* acid silica is separated by treatment with strong sulphuric acid; hydrofluoric acid is added and volatilized by heat along with the silica and sulphuric acid. Pure vanadic acid remains.

If *titanium* and *tantalum* are present, the substance may be mixed with sugar, carbonized by heat, and ignited in a stream of dry chlorine gas. The three chlorides distill over, and are received in water. Sulphuric acid is added, which precipitates silica and tantalic acids, leaving titanic acid in solution. The precipitate, washed and dried, is ignited, and digested with hydrofluoric acid in a platinum dish. Silica dissolves; tantalic acid remains.

From *phosphoric acid* silica is separated by the general methods indicated above. After the silica has been rendered insoluble, the phosphoric acid is found in the filtrate, and determined in the ordinary manner.

§ 94. ALUMINATES.

Emery, Sapphire, &c.—Place the powdered emery in a large platinum crucible, with eight times its weight bisulphate of soda, and ignite until perfect decomposition and solution is obtained. The mass when cool is treated with water containing a little sulphuric acid; the whole heated, when all dissolves, except silica. Any portions of the mineral which may have escaped decomposition, and which are detected by their grittiness, are filtered off, washed, and once more ignited with bisulphate of soda, and the result added to the rest. The substances present are determined by the methods already laid down.

§ 95. BORON AND BORACIC ACID.

When boracic acid exists in solution, unaccompanied

by any other substance, except ammonia; it is determined by adding a known excess of fused carbonate of soda, which is dissolved in the liquor, the whole evaporated to dryness, ignited, and weighed. A weaker heat must never be employed after a stronger one. The carbonic acid in the fused mass is now estimated. If the amount of soda in the carbonate originally used, and the carbonic acid which has escaped during the process be subtracted from the weight of the fused mass, the quantity of boracic acid will be most accurately obtained.

From bases, boracic acid is separated by means of a mixture of sulphuric and hydrofluoric acids; the fluoride of boron escapes on applying heat to redness. The boracic acid is estimated as loss. The bases remain as sulphates.

Boracic and phosphoric acids may be separated by precipitating the latter as ammonia-phosphate of magnesia.—*Rose*.

From silicic acid, in presence of bases, boracic acid is separated on decomposing with muriatic acid, when the silica is of course rendered insoluble.

If the substance be undecomposable by acids, the compound, in fine powder, is ignited with carbonate of soda, the mass extracted with hot water, and the filtrate precipitated with carbonate of ammonia to remove any traces of alumina and silica which have been taken up. The liquid is evaporated to dryness, sulphuric acid poured on the dry mass, and the boracic acid dissolved in alcohol.—*Gmelin*.

§ 96. SELENIUM.

Solutions containing selenious acid are estimated as tellurium by means of an alkaline sulphite. The reduced selenium is very carefully dried, and weighed on a balanced filter. Substances containing selenic acid are fused in a porcelain crucible with a little nitre, the mass dissolved in water, the solution mixed with nitric acid, nitrate of baryta added, the precipitate washed, dried, ignited, and weighed. 141 parts of this salt represent 64 of selenic acid, or 40 of selenium.

Selenium being precipitated by HS from acid solutions, and its sulphuret being soluble in excess of hydrosulphate of ammonia, its separation from the alkalies, earths, and the majority of metallic oxides, is very simple. Or, the powdered seleniuret may be oxidized by fusion with carbonate and nitrate of potash or soda, as directed for metallic sulphurets (I. § 24), and the selenic acid determined as above. Compounds containing arsenic may be oxidized in the same manner, or by nitric acid, or nitro-muriatic acid; in which latter case carbonate of soda is added in excess, the whole evaporated to dryness, and fused. It is then redissolved in water with nitric acid, and precipitated with nitrate of baryta. The arseniate of baryta is retained in solution by the nitric acid. From mixtures of tin, antimony, and arsenic, selenium is separated by dissolving in muriatic acid, diluting with water after adding tartaric acid, and precipitating the selenic acid as seleniate of baryta. Heat must not be applied.

If selenic and sulphuric acids exist together, they are precipitated jointly by means of nitrate of baryta, and the precipitate is dried, ignited, weighed, and then heated in a stream of hydrogen gas. The sulphate of baryta remains untouched, whilst the seleniate is reduced to seleniuret, and may be dissolved out by muriatic acid. Selenic acid and seleniates are reduced to selenites, if digested in muriatic acid.

§ 97. SULPHUR.

The estimation of sulphur in alkaline, earthy, and metallic sulphurets, is described in I. § 6; that of sulphates, soluble in water or acids, in I. § 2; the separation of alkaline sulphates, sulphites, hyposulphites, and sulphurets, in I. § 7.

Sulphuric acid, combined with *lead*, *baryta*, or *strontia*, is thus determined: a weighed portion, in fine powder, is fused in a platinum crucible with three parts carbonate of soda. If lead is present too strong a heat should be avoided. The mass when cold is extracted with water, the sediment (containing the bases) filtered, washed, treated with nitric acid, and in the solution the baryta, strontia, and lead separated as usual. The sulphuric acid is determined in the aqueous solution where it exists combined with soda.

§ 98. FLUORINE.

Compounds of fluorine are generally determined by heating with sulphuric acid till all the fluorine and the excess of the sulphuric acid are expelled. The

fluorine is then estimated as loss. This operation must be performed in a platinum vessel.

To determine the water of crystallization, another portion is mixed with about six or seven parts of litharge in a small glass retort, previously weighed, and very strongly heated. The loss of weight indicates water of crystallization.

Fluorine may be thrown down from solutions as fluoride of calcium, by adding chloride of calcium. The solution, if acid, should be neutralized with carbonate of soda before adding the chloride of calcium. The whole is then boiled, the precipitate filtered, calcined, freed from carbonates by digestion with acetic acid, evaporated to dryness, digested again in water, filtered, &c., ignited, and weighed. 38·8 parts fluoride of calcium contain 18·8 of fluorine.

Fluorides are separated from *phosphates* by expelling the fluorine by means of sulphuric acid.

From *silicates* fluorides are separated by igniting for a long time in fine powder, with four parts of carbonate of soda. The mass is treated with water, filtered, carbonate of ammonia added to the filtrate, which throws down traces of silica and alumina. This precipitate, along with the original residue, insoluble in water, is treated in concentrated and muriatic acid. The silica separates in a jelly. The liquid containing it is evaporated to dryness, cooled, moistened with muriatic acid, and water added. The silica is filtered off. The filtrate contains the bases.

Fluoride of silicon exists still in the liquid to which carbonate of ammonia was added. It is concentrated

in a platinum capsule, acidified with muriatic acid, and let stand in the cold, covered with filter paper, for twenty-four hours; towards the end of the time the liquid may be slightly warmed. The liquid is then mixed with an excess of pure ammonia, free from carbonic acid, poured into a stoppered flask, chloride of calcium added, and the flask closed. When the precipitate (fluoride of calcium) has settled, the clear liquid is decanted off, replaced by water which has been boiled, and cooled in a closed vessel, and the flask is stoppered again. When the precipitate has settled again, it is thrown upon a filter, washed, ignited, and weighed.

If *phosphoric acid* and *alumina* are present, decompose by ignition with carbonate of soda and silica, the quantity of the latter added having been most exactly weighed. Dissolve in water, wash the insoluble residue; add to the filtrate some carbonate of ammonia, which throws down the rest of the silica. This is filtered off, and chloride of calcium added; the ignited precipitate is treated with acetic acid to remove carbonate of lime, and the undissolved residue filtered off, washed, &c., ignited, and weighed. It is then treated with strong sulphuric acid in a large platinum crucible, at a gentle heat, till all fluorine is expelled. The residue is treated with alcohol, which dissolves out phosphoric and free sulphuric acids. Sulphate of lime remains undissolved, and is weighed. The phosphoric acid is freed from alcohol by heat, and determined by sulphate of magnesia and ammonia. Subtracting the phosphoric acid, and the lime weighed

as sulphate, from the weight of the precipitate, the residue is fluorine.

§ 99. CHLORINE. (See I. § 2.)

Volatile chlorides are dissolved in water before their estimation with nitrate of silver. Any matter deposited on decomposition is filtered off, and a little nitric acid is added. To weigh very volatile chlorides, some of which evolve heat, and occasion loss when mixed with water, it is best to take a very thin bulb of glass with an aperture drawn out in a long fine point. This bulb is weighed, heated to expel air, its point plunged into the chloride, which ascends into it. The bulb is then again weighed, with its contents, put into a stoppered bottle containing water, which is shaken till the bulb breaks.

From chrome chlorine is separated by diluting with water, neutralizing with ammonia, and acidifying with nitric acid before adding the nitrate of silver.

Chlorine in presence of the acids of *phosphorus* and *arsenic* is determined in the usual manner, a sufficiency of nitric acid having first been added.

If soluble *sulphurets* or *hyposulphites* be present along with the chlorides, a little sulphuret of silver will be thrown down along with the chloride. It should be digested with strong nitric acid until it becomes white.

If *selenium* is present the liquid, after being mixed with water, is digested for a considerable time before the deposit of selenium is filtered off. The filtrate is then considerably diluted, a great excess of the nitrate

of silver is avoided, the clear liquid is poured off from the precipitate as soon as it settles, and replaced by nitric acid. The whole is then heated, the acid poured off and hot water added as long as the liquid is rendered turbid by muriatic acid.

Solutions containing *tellurium*, after being mixed with water and filtered, are treated in the same manner; the chloride of silver being easily freed from tellurium by treatment with nitric acid.

Chlorides of *tin* are first treated with HS in excess, and the liquid allowed to digest in a stoppered flask till all sulphuret of tin is deposited. Filter, add a little sulphate of copper to remove any excess of HS; filter again, and add nitric acid and nitrate of silver as usual.

From solutions of the chloride of *titanium* titanous acid is thrown down by ammonia; set aside till the excess has evaporated, filtered, and the filtrate treated as usual.

To solutions of *antimony* tartaric acid is added, and the antimony removed with the precautions given for tin.

Chloride of *molybdenum* is treated like chloride of selenium. In chloride of tungsten the chlorine is determined merely as loss.

The chloride of *silver* and the subchloride of *mercury* being insoluble in water and nitric acid are otherwise determined. Chloride of silver is reduced in a stream of hydrogen at a gentle heat, and the chlorine either determined as loss, or led into a solution of ammonia and determined there. Chloride of

lead may be treated thus, avoiding a high temperature. Chloride of silver may also be reduced by digesting with a slip of clean zinc, a few drops of sulphuric acid being added. The chloride of zinc in the filtered solution is then determined in the ordinary manner.

Subchloride of mercury is decomposed by digestion in pure soda, the filtrate acidified with nitric acid, and the nitrate of silver then added.

Most metallic chlorides may be decomposed, if required, by treatment with saturated HS water or hydrosulphate of ammonia in a stoppered flask.

All vessels in which chloride of silver is being digested, filtered, or dried, should be preserved as far as possible from the action of daylight, and should never be exposed to the rays of the sun.

§ 100. OXYGEN COMPOUNDS OF CHLORINE.

Perchloric acid is determined as perchlorate of potash. (I. § 2.)

Chlorates are ignited carefully, and the chlorine in the residual chloride determined.

Chlorides may be separated from chlorates and perchlorates by adding nitrate of silver to the aqueous solution. No nitric acid is added. The chlorates and perchlorates remain in the filtrate.

§ 101. CHLORINE FROM HYDROCHLORIC ACID.

Dissolve a weighed portion, dissolve sulphate of potash in the liquid, and expel the free chlorine by evaporation *in the dark*. Then, when the liquid is

found to have no bleaching effect on vegetable colours, determine the combined chlorine with nitrate of silver as usual.

Determine the entire chlorine in a second sample; the excess of the second determination over the first shows the amount of *free* chlorine.

§ 102. BROMINE.

Bromine is determined, whether existing alone or in compounds, exactly as chlorine. The bromides are decomposed in the same manner as the corresponding chlorides. Bromide of silver contains 42.06 per cent. of bromine.

Bromides are thus separated from chlorides. The mixture is precipitated with nitrate of silver, as if a chloride alone were present; the mixed precipitate is dried, ignited, and weighed as usual. The crucible is then again warmed until the bulk of the mass can be taken out with a glass rod. A weighed portion of this is heated in a current of dry chlorine gas, which expels the bromine. The loss of weight, multiplied by 1.826, gives the amount of bromine expelled. The weight of the silver existing in the pure chloride obtained last, *plus* the calculated amount of bromine is deducted from the mixture of bromide and chloride of silver. The remainder is the chlorine; or—

Reduce the mixed precipitate with dilute sulphuric acid and zinc; filter; add baryta in slight excess; filter again, and evaporate the filtrate to dryness. Treat with absolute alcohol. Bromide of

barium dissolves, whilst the chloride of the same base remains untouched.

§ 103. IODINE.

Iodine is determined, and the iodides decomposed exactly as chlorine and bromine. Iodide of silver contains 53·85 per cent. of iodine.

From bromine and chlorine iodine is separated by adding to the solution a soluble salt of the protoxide of palladium. The mixture is allowed to stand for some time; the black precipitate washed with boiling water and dried for twenty-four hours under the air-pump. It contains 70·28 per cent. of iodine.

Iodine may also be separated from chlorine by adding a solution of protosulphate of copper saturated with sulphurous acid. The iodine is entirely thrown down as subiodide of copper. It contains 66·32 per cent. of iodine.

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PART II.

INTRODUCTION.—VOLUMETRICAL ANALYSIS.

VOLUMETRY is an analytical procedure depending not upon weight but measure. A *standard* test solution (*i.e.* of known strength) is dropped from a graduated pourette into the solution of the sample in question, until a certain effect, as, *e.g.*, a change of colour is produced. By observing how many degrees have been employed in comparison with the number required to produce the same effect upon an absolutely pure specimen, we can easily calculate how much of the substance to be determined is present in our sample. Thus, suppose we have a test-liquor of such strength that 100 degrees are requisite to produce some given change in the solution of 10 grains of pure iron, and that in operating upon 10 grains of an iron ore, only 65 degrees are needed to give the same effect, we perceive that the 10 grains of ore must contain $6\frac{1}{2}$ grains of actual iron, or, in other words, that the ore contains 65 per cent.

Volumetry is in most cases somewhat inferior in accuracy to the ordinary analytical procedures, and cannot serve to determine all the constituents of a complex body. Hence it is best fitted for the examination of commercial samples whose value

depends upon the proportion of some one ingredient. In such cases the result may be obtained with an accuracy sufficient for all practical purposes, and with great economy of time. This advantage is most striking when certain determinations are of very frequent occurrence.

The apparatus used in volumetry is very simple. Graduated pourettes, or Mohr's alkalimeters, serve to register the quantity of test-liquor consumed. The operator commences by weighing out and dissolving a sample as in ordinary analysis. The solution is placed in a beaker or tall glass jar, and the test-liquor gradually and carefully added until the required effect be produced. Great attention must be paid to the purity of the materials from which the standard test-liquors are prepared, and their value, or saturating power, must be determined by a previous trial.

Results of tolerable accuracy may also be obtained by the *colorimeter*. In this procedure the colour of the dissolved sample is compared with that of a normal solution, observing how much the latter requires to be diluted with water before it exhibits the same intensity of tint as the solution of the sample. The solutions to be compared are placed in test-tubes of equal calibre, and held up against a sheet of white paper. An instrument which admits of greater accuracy may be made by cutting off from a stout glass tube, about 1 to $1\frac{1}{4}$ inch in diameter, a number of hoops or segments. These are ground level at one edge, and then firmly cemented down upon a slip of stout plate-glass. The whole are then applied to the

grindstone together, and are thus reduced into cells exactly equal in size. The segments should be cut of such a width that, after grinding, the cells may have a depth of about half an inch. To use this colorimeter the cells are filled with the liquids to be compared, and closed by covering them with a slip of plate-glass similar to the one on which they are fastened.

§ 1. ALKALIMETRY.

1. *Soda-ash*.—Prepare a standard solution of sulphuric acid, such that nearly 1000 grain measures may be consumed in neutralizing 38·8 grains of actual soda.

To ascertain the exact saturating power of the dilute acid, it is necessary to prepare a sample of absolutely pure carbonate of soda.

For this purpose select the smallest well-defined crystals of commercial carbonate, dissolve them in water, concentrate, and when the solution is near crystallizing, cool rapidly, and stir it continuously, so that the carbonate may be deposited in a fine crystalline meal. Pour away the mother-liquor, wash with pure water, and repeat this process till the washings, after acidulation with nitric acid, are no longer affected by nitrate of silver or chloride of barium. Place the mass in a basin of platinum, silver, or clean sheet iron, dry, and expose for two or three hours to a high sand-bath heat, not amounting quite to redness. Dissolve and filter off the deposit of silica. Evaporate

again to dryness, adding from time to time fragments of carbonate of ammonia. Redissolve and filter once more; if any flakes of silica appear, evaporate to dryness, and heat in platinum for three hours to a temperature just bordering on dull redness. Let it cool under a bell-jar along with sulphuric acid or chloride of calcium, and preserve it in well stoppered bottles.

Weigh out 50 grains of this carbonate of soda, place it in a porcelain basin capable of holding about a pint, dissolve in water, and set the basin over a gas flame. Meantime cut up some of the very palest neutral-grey litmus paper into small bits, and lay them on a white slab close at hand. Fill a Mohr's alkalimeter with the standard acid, and let it drop into the alkaline solution, stirring vigorously, and maintaining a heat close upon boiling. From time to time a drop of the solution is applied, by means of the stirring-rod, to one of the bits of litmus paper. As soon as the faintest tint of *permanent* red appears the operation is at an end, and the number of degrees on the alkalimeter consumed are read off. The only difficulty in this process is distinguishing between the true red caused by a very minute excess of sulphuric acid, and the fugitive purple stain occasioned near the end of the operation by free carbonic acid. If 75.5 degrees have been consumed, then the acid has the exact saturating power specified, viz., 1000 grain measures = 38.8 grains actual soda. Suppose, however, that only 74 degrees have been used, then label the bottle of test acid, "74 degrees = 29.3 grains of

actual soda," the amount contained in 50 grains of pure carbonate.

In testing commercial soda-ash, weigh out 50 grains of the sample and proceed exactly as above. Suppose that 70 degrees of the alkalimeter are consumed, then $74:70=29.3:x$, amount of actual soda in 50 grains of the sample.

If a sample of ash contains alkaline sulphites, it is well to use *violet paper* instead of litmus, and to consider the process at an end when the paper takes a yellow tint, indicative of the liberation of sulphurous acid.

The test acid, of which a large stock should be prepared, where alkalimetical operations are frequent, should be free from sulphurous and nitrous acids.

With samples of caustic soda the procedure is exactly similar.

2. *Potash*.—1000 grain measures of an acid capable of neutralizing 38.8 grains of pure soda will neutralize 58.66 grains of pure potash.

Supposing that 74 degrees of an acid neutralize 29.3 of soda, it will correspond to 44.3 of actual potash. Proceed, therefore, as above, and calculate—

$$74:n \text{ (i.e. number of degrees consumed)} = 44.3:x.$$

3. *Ammonia*.—74 degrees of the acid neutralizing 29.3 of soda will correspond to 15.96 of pure anhydrous ammonia (NH^3). Proceed as above, and calculate—

$$74:n = 15.96:x.$$

In examining samples of caustic ammonia, take 50, or any other known number, *grain measures*, and

dilute with water before adding the test acid. The application of heat is unnecessary.

Additional Remarks.

4. To determine the amount of *carbonic acid* in a sample of alkali, caustic, carbonate, or bicarbonate, proceed as in I. § 8, *b*.

5. To ascertain the amount of *caustic alkali* present along with *carbonate* in soda-ash or potashes, see I. § 5; or—

Determine the whole amount of alkali in, *e.g.*, 50 grains with test acid. Then take another sample of 50 grains, add to its solution chloride of barium in large excess, filter, wash slightly, and again determine the alkali in the filtrate with test acid. This second determination shows the amount of caustic alkali, and this subtracted from the former gives the carbonate.

6. If *sulphurets*, *sulphites*, and *hyposulphites*, are present in soda-ash, add a little pure chromate of potash.

If *earthy carbonates* are found in an alkali, the sample before testing must be dissolved in hot water, filtered, and the filter paper thoroughly rinsed with hot water.

7. *Moisture* in a sample of caustic or carbonated alkali is determined by heating 200 grains in a platinum crucible for two hours almost to redness, and cooling over sulphuric acid. At a red heat soda-ash loses a little of its carbonic acid. Potash should be heated in a crucible with a well-fitting lid, and which is closed during weighing. *Bicarbonates* are dried over

sulphuric acid as long as any change of weight is observed.

8. *Neutral carbonates* may be detected in bicarbonates by adding a solution of chloride of mercury, which gives an orange-red precipitate with the former.

9. *Vat waste* (from alkali works).

Weigh out 1000 grains of the moist waste, dry, and determine loss of weight.

Weigh out a second portion of 1000 grains, also in the moist state, extract with 6 ounces boiling water, filter, and wash slightly with boiling water. Pass through the filtrate a current of carbonic acid gas as long as any smell of HS is perceived, boil, filter, and determine alkali in the clear liquid. The determination of moisture will enable the operator to calculate the proportion of alkali in the dry mass.

10. *Soda in Potash*.—Mix half an ounce of the sample with water, add sulphuric acid in excess, evaporate to dryness, ignite, and weigh the residue. Agitate the powdered saline mass in a graduated cylinder with six times its weight of a saturated solution of pure sulphate of potash. The clear liquid is drawn off with a syphon, and the same amount of a saturated sulphate of potash solution is again applied to the residue. After some time the residue is poured upon a weighed filter, the funnel covered during filtration, the filter, when the liquid has drained off, is weighed while moist, and then again when dry, at 212° Fahr. The difference is the evaporated water of the solution of sulphate of potash, and as the degree of concentration of this solution was known, the quantity of the salt

dissolved in this evaporated moisture is calculated, and subtracted from the weight of the saline residue. If the sample of potash was free from soda, the weight of the sulphate of potash, after the above-named deduction, should be the same as just after ignition. If soda was present a loss will be observed. Let this loss = p , then—

$71.2 : 53.2 = p : x$, the amount of dry carbonate of soda in the sample.

11. *Common soda*, sold especially in the clothing district of Yorkshire, as distinct from soda-ash (*vulgo*, Newcastle or Scotch soda) and soda crystals, is made by dissolving salt cake, and adding to the liquid either ground chalk or cream of lime, or both. Double decomposition takes place to a certain extent, and the clear liquid, more or less perfectly separated from the precipitate of lime, is boiled down to dryness.

To examine it, weigh out 100 grains, dissolve in water, filter off any insoluble matter, and proceed as for soda-ash. The amount of alkali is very much smaller.

12. *Washing or scouring sugar*, or *soap-ash*, is generally made of soda-ash, sometimes even common soda, ground up with a small quantity of palm oil, or sometimes with yellow rosin. Common salt, sulphate of soda, or any other cheap saline matter which is at hand is added in abundance, as also a little turmeric to colour. The whole has the appearance of coarse moist sugar.

Examine as for soda-ash, carbonate of soda being the only article of value present. On dissolving a

sample in water, rosin, oil, and turmeric will rise to the surface, and may be easily recognized.

13. *Washing powders* or *washing crystals* generally consist of carbonate of soda deprived of its water by heat under constant stirring. Some are let down with common salt, and many are "blued" with a little smalt or artificial ultramarine. Examine as in II. § 1.

Some consist of soda-ash mixed with sufficient lime to render the soda caustic, when the whole is boiled in water. To determine the amount of alkali, weigh off 50 grains extract in boiling water, and proceed as for vat waste (II. § 1).

14. *Washing pastes* consist of caustic soda thickened with some farinaceous matter. Test a small portion for carbonic acid. If it is present proceed as in I. § 5; otherwise as in II. § 1.

15. *Washing liquor* is simply an impure and dilute caustic ammonia (II. § 1).

16. *Urine* is an article of commerce in the clothing districts, being used for scouring wool; its value depending solely on the ammonia which it contains. It is examined like liquid ammonia. It is largely adulterated with water and common salt.

17. *Urine substitutes* are merely washing crystals or powders, and are examined as soda-ash.

§ 2. AMMONIACAL SALTS.

Moisture is determined by drying a weighed portion over sulphuric acid.

Fixed salts (common salt, gypsum, sulphate of soda) often occur to a serious extent in commercial samples

intended for agricultural uses, and are easily determined by igniting a weighed sample in a porcelain crucible, the heat being raised very gently at first to prevent loss by decrepitation.

Chloride of iron is often found in salammoniac, which is then unfit for the preparation of mordants and colours. It is determined by adding liquid ammonia in excess to the solution, collecting and weighing the precipitate of sesquioxide of iron.

§ 3. ACIDIMETRY AND COMMERCIAL ACIDS.

1. To estimate the real saturating power of a commercial sample of acid (sulphuric, nitric, hydrochloric, or acetic), we require first a standard alkaline solution. This may be made by dissolving 270 grains of the pure carbonate of soda (II. § 1, 1), in 10,000 grain measures of water. 1000 grain measures of this will exactly saturate 20 grains of dry sulphuric, 18.5 grains of dry hydrochloric, 27 grains of dry nitric, and 30 of crystalline acetic acids.

Weigh out, by means of a dropping tube, 50 grains of the sample to be tested into a beaker, dilute with water, and add the test-liquor exactly as in alkalimetry, until a drop no longer reddens litmus paper. A simple calculation shows the amount of actual acid in the sample.

An ammoniacal test-liquor may also be employed; 1000 grain measures of liquid ammonia of specific gravity 0.992 saturating exactly twice the quantity given above of each acid.

Additional Remarks.

2. *Sulphuric acid* or *oil of vitriol* for the use of the colour-maker, dyer, calico-printer, and electro-plater, should be absolutely free from compounds of nitrogen. To detect these drop into a portion of the acid a fragment of protosulphate of iron. If nitrogen compounds are present, a reddish colouration appears in time more or less intense in proportion to the amount of the impurity; or—

Throw in a small quantity of iron filings. A rose, bluish-red or violet colour is produced if nitric or hyponitric acids be present; or—

Add a little infusion of cochineal. Its colour is destroyed by the oxides of nitrogen. A similar result takes place with sulphate of indigo; or—

Heat the suspected acid in a test tube or small retort with a few iron filings, and pass the gas which is given off into a little tincture of guaiacum. If nitrogen compounds are present the tincture will be instantly turned blue.

Arsenic, which often occurs to a large extent in samples of sulphuric acid prepared from iron and copper pyrites, unfits it for pharmaceutical, and even for agricultural purposes. It is detected by passing HS through the dilute liquid, when a yellow precipitate will fall.

3. *Hydrochloric* or *muriatic acid* often contains iron (perchloride) and sulphurous acid, both of which are injurious in dyeing and mordant making. Iron is

detected and estimated by adding to a known quantity of the acid an excess of ammonia. The whole is allowed to stand for some time filtered, and the precipitate washed, dried, ignited, and weighed.

To detect sulphurous acid dissolve a little zinc in the sample of acid, and pass the gas evolved through a solution of litharge in caustic alkali. If sulphurous acid be present the solution will be blackened.

4. *Nitric acid* or *aqua fortis* is often contaminated with sulphuric and muriatic (hydrochloric) acids. Both are readily detected by the common tests, namely, chloride of barium in a diluted portion for sulphuric acid, and nitrate of silver in a similarly diluted portion for muriatic acid. Pure nitric acid diluted with water should give a precipitate with neither.

5. *Acetic acid* may contain as impurities sulphuric acid—add hydrochloric acid and chloride of barium; hydrochloric acid—add nitric acid and nitrate of silver; oxalic acid—add ammonia and chloride of calcium; and in all three cases filter off the precipitate which will appear if the impurity be present. Free sulphuric acid may also be detected by dissolving 1 part of sugar in 30 of water, and evaporating to dryness at a steam heat in a porcelain capsule. A drop of the liquid to be tested is then let fall into the capsule, which is then again heated to 212° Fahr. If sulphuric acid be present a blackish or greenish brown spot will appear where the drop fell.

This process is commonly used to detect free sulphuric acid when present along with solutions of sulphates. It must be remembered, however, that

concentrated solutions of alum, and probably also of other sulphates of feeble bases, give the same reaction with sugar, requiring only rather more time.

6. Alkaline and earthy salts are often added to a large extent to the acids of commerce; in which case a great discrepancy exists between the saturating power of an acid, and its specific gravity. This fraud may be readily detected by evaporating one ounce of the suspected acid to dryness at the lowest possible heat, when the saline matter will remain.

Sulphuric acid added to nitric (a common fraud) may be detected in the same manner, for when all the true nitric acid in a sample has been evaporated off, the sulphuric present remains as a more oily liquid, not volatile at the temperature at which the nitric acid evaporated. The quantity of the adulteration may thus be roughly estimated in a short time and with little trouble.

7. Nitric, muriatic, and acetic acids may likewise be conveniently estimated by means of carbonate of baryta. For this purpose a known excess of that substance, say 150 to 200 grains, is weighed off, and put in a beaker. 100 or 50 grains of the acid are then weighed off, diluted, and poured upon the carbonate. The whole is allowed to stand at a gentle heat for some time, until all effervescence, even on stirring, has ceased. It is then filtered, washed, dried, &c., and the residue weighed. The loss in weight shows the amount of carbonate of baryta which the acid has saturated and dissolved; whence its strength is easily calculated.

§ 4. SALT CAKE.

Residue on decomposing common salt with sulphuric acid in the manufactures of artificial soda and muriatic acid.

Salt cake contains about 94 per cent. sulphate of soda, on which its value depends; besides small quantities of free sulphuric acid, sulphate of iron, and undecomposed common salt.

For its commercial analysis the following process will serve :—

a. Weigh out 100 grains, and heat to dull redness in a platinum capsule for some time, avoiding fusion. Weigh again when cold. The loss indicates free acid, and the acid contained in the sulphate of iron.

b. Dissolve the residue from the last operation in water. Filter, wash, dry, ignite, and weigh the insoluble matter, sand, and oxide of iron, from the decomposed sulphate.

c. To the filtrate and washings from No. 2, add a little pure chromate of potash, and drop in from a pourette a standard solution of nitrate of silver. The moment that a permanent yellow tinge appears in the precipitate, the operation is completed.

From the amount of nitrate of silver consumed, the quantity of chloride of sodium present in the sample is calculated. (100 grains of nitrate of silver correspond to 34.45 grains of chloride of sodium, or common salt). The amount thus found being added to the loss in *a*, and the residue in *b*, and the sum subtracted from 100, the remainder is sulphate of soda.

Instead of adding the chromate of potash to the saline solution, drops of this reagent may be spotted upon a white slab, and a drop of the solution may be added to one of these from time to time as the operation proceeds, ceasing as soon as a drop exhibits a yellow or orange shade.

This method of determining chlorine is applicable in solutions of common salt, urines, mineral waters, nitre, potash, soda-ash, &c. Chlorides of barium, calcium, iron, mercury, &c., are previously precipitated with pure carbonate of soda; the chloride of sodium thus formed containing all the chlorine previously present in the chloride of barium. Indirectly, it may be also applied to the other substances stated I. § S, *a*. The solution to be tested may be neutral, or contain a slight excess of carbonate of soda.

Nitre cake, the residue from the manufacture of nitric acid, is examined in a manner precisely similar. It is necessary to ascertain whether potash- or soda-saltpetre have been employed.

§ 5. NITRE.

Nitrate of Potash, Potash-saltpetre.

The value of this salt may be determined either by ascertaining the amount of its impurities (technically styled *refraction*), or by determining the proportion of real nitre present.

1. In proceeding by the former method we determine—

a. Moisture.—Dry 1000 grains at 212° Fahr., as long as any loss of weight is perceptible.

b. Dissolve 100 grains in water; filter, wash, dry, and weigh the residue.

c. Divide the filtrate and washings of *b* in two equal parts.

aa. Determine in one of these parts the *chlorine* present as chloride of sodium.

bb. In the other half determine the sulphates. The remainder should be nitrate of potash. This method is, however, deficient in accuracy.

For samples of *soda-saltpetre* (cubic nitre), it may be used with more confidence.

2. The direct determination of the nitric acid in nitrate is thus conducted—

a. Take a tubulated long-necked retort, containing about 3000 grain measures; fix it with the neck sloping a little obliquely upwards. Into its body are put about 25 grains (accurately weighed) of the purest pianoforte wire, and about 700 grain measures of pure concentrated muriatic acid are added. A current of hydrogen gas, washed in a solution of potash, is passed into the retort through the tubulure, by means of a glass tube dipping only about three-fourths of an inch into the retort. The neck of the retort is united to a **U**-tube containing a little water. The body of the retort is heated on the water bath till the iron is completely dissolved. It is now allowed to cool in the current of hydrogen, which is afterwards strengthened. The tube connecting the neck of the retort to the **U**-tube is disengaged, and the sample of nitrate (which should not contain more than three grains of nitric acid) having been weighed

in a small tube, is allowed to slide down the neck of the retort into the body. The retort is then again connected to the U-tube, and heated on the water-bath for about a quarter of an hour. The water-bath is then removed and a lamp applied, so as to make the contents of the retort boil briskly, and losing their original dark colour, assume the hue of perchloride of iron. After this point is reached, the boiling is still continued for a few minutes. Whenever the retort is moved or shaken, care must be taken that nothing is deposited on its sides. Before removing the lamp the current of hydrogen is strengthened, to prevent the entrance of air through the U-tube. The retort is allowed to cool in the current of gas, and the contents are much diluted with water.

A solution of permanganate of potash must have been previously prepared, and its value ascertained as follows:—Dissolve 25 grains of the purest iron wire in 1500 grain measures of pure hydrochloric acid, without the application of heat. When the whole is dissolved dilute with about a pint and a half of water. Now fill a pourette with the permanganate solution and drop it into the iron solution until it ceases to be decolorized, and a very faint pink tint appears in the liquid. Note the number of degrees required to produce this result. The permanganate is of a convenient strength if 50 degrees have been consumed, in which case each degree of the pourette answers to half a grain of metallic iron.

The contents of the retort being, as was said, largely diluted with water, the permanganate solution is

added from a pourette, until a pink tinge appears in the liquid. The number of degrees employed for this purpose shows the quantity of iron remaining in the state of protochloride. The remainder of the iron has, of course, been transformed into perchloride by the nitric acid of the sample employed, and as 30.88 grains of iron require for this purpose 18.76 grains of pure nitrate of potash, the amount of actual nitrate present in the sample is readily obtained.

The same process applies to the nitrate of soda; or—

b. Take a porcelain crucible one inch and a half wide at bottom, and the sides about one inch high, with a well fitting lid. Ascertain its weight, and introduce from 7 to 15 grains of the sample to be tested; put on the lid and heat until it fuses smoothly and without bubbles. The loss of weight is water. It is needful that the nitre be spread out evenly on the bottom of the crucible. Sulphate of lead, dry and powdered, is now spread upon the nitre, amounting to eight or ten times its weight. The crucible is again weighed and placed over the lamp. As soon as orange vapours cease escaping, which will be in about ten minutes, the process is at an end. Weigh after cooling. The loss of weight is equal to the nitric acid contained in the sample acted upon.—*Zenner.*

3. To detect the presence of *nitrites* in nitrates, salt cake, &c., proceed as follows:—

a. To a solution of the suspected substance, or to the liquid, as the case may be, add one or two drops

of solution of yellow prussiate of potash, not enough to communicate a perceptible yellow tint. A few drops of acetic acid are then added, and immediately, or in some minutes, according to the quantity of nitrite present, the liquid assumes a rich golden tint.

It is better, when testing for minute quantities, to use two similar vessels, one containing pure water, and the other the liquid under examination; to both of which the reagents are added in precisely equal quantities. The vessels should be equally exposed to light, with a sheet of white paper behind them.

This may be converted into a test for the *nitrates*, by boiling the sample for a few minutes with clean shavings of lead, and proceeding as above; the absence of nitrites in the substance having been first ascertained. Lead reduces even the nitrate of potash to nitrite.—*Schæffer*.

b. A few drops of a dilute aqueous solution of iodide of potassium (free from iodate) are mixed with starch paste, and then dilute hydrochloric acid of specific gravity 1.006 is added. The liquid suspected to contain the nitrite, if alkaline, is first acidulated with a little hydrochloric acid, and then added to the test-mixture, when, sooner or later, a violet tint makes its appearance. The experiment is best performed in a porcelain capsule. With $\frac{1}{5,000,000}$ of nitrous acid, a violet tint appears in about fifteen minutes.

This test may be made applicable to nitrates in the same manner as the above.—*Price*.

Nitric acid, and the nitrates may also be detected

by the following method:—To the solid or liquid supposed to contain nitric acid in a free or combined state, add a few drops of a very strong solution of yellow prussiate, and then some pure hydrochloric acid, mixing the ingredients well together, both before and after the addition of the acid, and gradually raise the temperature of the mixture to 160° Fahr., or a little higher; let it cool, and then neutralize with carbonate of soda, a slight excess being of no consequence. Filter, if there be much precipitate, and finally add to the fluid a drop of either sulphuret of ammonium, potassium, or sodium, when, if nitric acid was present, a fine purple colour will be produced which soon disappears. In using this test, much dilution of the materials should be avoided. The hydrochloric acid should be of specific gravity 1.15; a weaker acid not giving satisfactory results. If the nitric acid or nitrate be in very minute quantity, the mixture of it with the prussiate and acid should be allowed to cool down to about 80° Fahr. before the carbonate of soda is added.

There is generally a striking difference between cases where no nitre is present, and where it occurs when the substance is heated with prussiate and hydrochloric acid. When nitric acid is not present, the mixture quickly becomes of a blue colour; but where nitric acid exists, it first becomes a yellowish green, and then an olive or dark brown.

In heating the mixture, the temperature stated should be maintained for a few moments until it ceases to acquire a darker shade.

§ 6. SULPHURIC ACID IN COMBINATION.

Dissolve 525.7 grains of pure, dry chloride of barium in 10,000 grain measures of water; 1000 grain measures of this will, of course, contain 52.57 grains chloride of barium, and will saturate 20.2 grains of dry sulphuric acid (SO^3). It is necessary to provide a slab of black glass, or a plate of common glass, beneath which is placed a sheet of black paper. Two bottles with elongated stoppers are also required, the one containing dilute sulphuric acid, the other a solution of chloride of barium. Call these bottles S and B respectively. Have at hand also, a number of small pieces of filter paper about an inch square. The sample in question—from 20 to 100 grains, according to its probable per centage of sulphuric acid—is now placed in a beaker, dissolved in water (or if needful, in an acid) strongly acidified with hydrochloric acid, and heated to boiling. The test-liquid is now dropped into it gradually, with vigorous stirring. From time to time fold one of the bits of paper into the shape of a filter, and holding it by one corner, place in it a few drops of the solution by means of the stirring-rod. Let two drops run through upon the black glass, and return the bit of paper with the rest of its contents into the beaker. The two drops should be pretty near each other, equal in size, and round, not spreading out into a splash; neither must they contain the slightest turbidity. To one of these spots apply a drop from S, and to the other a drop from B. If the latter

alone cause a white precipitate, more of the test-solution must be still added. If white precipitates are caused by both, that given by B still being the more dense and bulky, the additions must be made very cautiously, and test-drops taken more frequently, until both spots exhibit a perfect equality. The operation is now completed, the number of divisions on the pourette consumed is read off, and the amount of sulphuric acid calculated accordingly. A little experience is required to decide when the exact point of saturation be reached; and it is often useful after noting the number of degrees of test-liquid which have brought both spots to an apparent equality, to add a little more, and observe whether an excess begins to appear on the S side. It is likewise well, after adding the drops from S and B to the two spots, to wait for a short time before deciding, that the precipitate may have time to form.

§ 7. MANGANESE IN COMMERCIAL SAMPLES.

1. Treat a weighed quantity of the ore in fine powder with dilute nitric acid. *Earthy carbonates* are dissolved. Their amount is estimated as loss.

Another weighed quantity, air-dried like the former, is placed in a very small retort, to which is attached a bulb-tube filled with chloride of calcium and accurately weighed. A strong heat is then applied to the retort by means of the gas furnace. The chloride of calcium tube is then weighed, when its increase of weight shows the amount of *water* present in the sample.

The actual *peroxide of manganese* present in the sample may be thus determined:—Dissolve 113.53 grains of arsenious acid in potash, and then add hydrochloric acid until the solution amounts to 100 measures; one of these will correspond to one grain pure peroxide of manganese. Reserve this for use as a standard solution.

Next, standardize a solution of permanganate of potash as follows:—Take five measures of the above arsenious solution (corresponding to 5 grains of peroxide of manganese), dilute it with water, and drop the solution of permanganate in until all the arsenious acid is converted into arsenic acid; a change indicated by the appearance of a faint pink tinge in the liquid.

Take now 10 grains of the sample to be examined, put it into a small flask, and add 10 measures of the arsenic solution. Join to the mouth of the flask a Wills' nitrogen apparatus, half filled with a solution of potash. Apply a gentle heat to the flask until the manganese is dissolved. The contents of the flask are allowed to cool; they are then, together with the potash from the Wills' tube, poured into a larger flask, as also the rinsings of both vessels, and diluted with water. The amount of arsenious acid remaining unchanged is then determined by the permanganate solution. The quantity thus indicated being deducted from the number of measures of the arsenious solution employed at first, will give the value of the sample.

Thus, suppose 10 grains of the sample have been taken, and 10 measures of the arsenious liquid used,

and that the quantity of permanganate solution consumed, corresponds to 3·5 measures of the arsenious liquor, then $10 - 3\cdot5 = 6\cdot5$; or, the sample contains 65 per cent. of pure peroxide of manganese.—*Price.*

The arsenious acid must of course be dissolved in a small quantity of potash, so that the standard liquor, after adding the muriatic acid shall be powerfully acid.

2. Into the tube *a* of the apparatus I. § 8, *b*, we put 44 grains of the sample, previously well powdered and freed from any carbonates, by treatment with dilute nitric acid, and likewise 100 grains of the neutral oxalate of potash, also in powder. Sulphuric acid is then placed in the outer tube of the apparatus, the whole is weighed, and the experiment is conducted precisely as in the paragraph referred to. The loss of weight at the end of the experiment shows the proportion of pure peroxide in the sample; each grain of carbonic acid escaping, representing one grain of peroxide of manganese.

§ 8. SULPHUR IN COMMERCIAL SAMPLES.

Boil the finely-powdered sample in twenty times its weight of pure oil of turpentine, taking great care lest the vapours catch fire, decant off the liquid while hot, and dry and weigh the residual impurities.

See also I. §§ 24 and 25.

For the treatment of *sulphur ores* (iron and copper pyrites), see the same passages.

Traces of sulphur in minerals, as distinct from sulphuric acid, may be detected by fusing the sub-

stance with potash in a small clean iron crucible, or in a platinum spoon before the blowpipe. The mass is dissolved in a very little water and filtered, and a bright strip of silver foil placed in the liquid. If sulphur is present, the silver will sooner or later be blackened.

A more delicate test, especially for organic matter, is as follows:—Heat upon charcoal the substance in question, along with a little carbonate of soda by means of the blowpipe. The carbonate of soda may be advantageously mixed with a little starch to prevent oxidation of the sulphur. Put the fused mass into a watch-glass with a drop of water, and add a particle of nitro-prusside of sodium no larger than a pin's head. If sulphur is present in any form, a splendid purple will at once appear.

§ 9. GUNPOWDER.

1. Dry 200 grains over sulphuric acid, and note the loss of weight—*moisture*.

2. Treat another weighed sample with water, filter, wash, dry, and weigh the residue. The loss of weight = nitre. Test the filtrate, however, for common salt with nitrate of silver, and determine its amount, if present (I. § 1, *b*).

3. Boil the dried residue from No. 2, with a saturated solution of permanganate of potash, as long as the liquid is decolorized. Then acidify with muriatic acid, filter, wash, dry, and weigh the residue of carbon, and in the filtrate determine the sulphuric acid, from which the sulphur is calculated.

§ 10. BLEACHING POWDER.

To find the value of bleaching powder, *i. e.*, its amount of available chlorine, the following process is employed:—A test-liquor is prepared by dissolving 100 grains of pure arsenious acid in pure hydrochloric acid, applying a very gentle heat. Make up the solution to ten ounces, by adding distilled water. Every ounce of the solution contains of course 10 grains of arsenious acid.

To apply the test, weigh out 100 grains of the sample of bleaching powder, and rub it well in a porcelain mortar with a little water. Rinse it into a measuring-glass capable of holding 2000 grain measures, and add water enough to make up exactly 2000 grains. A pourette is now filled with this mixture which is first well stirred up. Every degree of the pourette contains one half grain of bleaching-powder.

An ounce of the solution of arsenious acid is now poured into a beaker and coloured a decided blue by the addition of a little sulphate of indigo; and the bleaching liquor is carefully dropped in from the pourette, stirring all the time, until the blue colour disappears.

The number of degrees required for this purpose contain exactly 7.17 of available chlorine. Each degree having contained one half grain of the powder, the amount of chlorine contained in 100 parts is readily calculated.—*Penot.* Or—

Prepare a test-paper by heating together 15 grains

iodine, 105 grains crystallized carbonate of soda, 45 grains potato starch, 10 ounce measures of water, until the liquid becomes colourless, when it is made up to 40 ounces with water. White paper is saturated with this liquid and dried.

The test-solution is then made by dissolving 100 grains of arsenious acid and 292.7 grains of crystallized carbonate of soda in 10 grains of water, and proceeding as before. The process is complete when a drop of the solution produces a blue colour upon the test-paper.

§ 11. BORAX, TINCAL, BORACIC ACID.

Determine in a commercial sample *chlorine* (I. § 1, *b*, and II. § 4, 3), which indicates common salt, and *alumina*, if present (I. § 14, *b*), 1.08 grains of alumina represent 10 grains of common alum.

The amount of soda combined with boracic acid may be determined alkalimetrically (II. § 1), 31.2 grains of soda represent 190.8 of *pure* crystalline borax.

In raw tincal note also the proportion of matter insoluble in water.

Boracic acid may be first dried at a very gentle heat and then raised to dull redness, noting the loss on each operation. The residue is treated with excess of alcohol, the insoluble matter (earthy salts, clay, sand, &c.), filtered off, washed with alcohol, dried, and weighed. This weight is added to the losses on drying and on igniting the sample, and the sum is subtracted from the entire weight taken for analysis. The remainder is actual boracic acid.

§ 12. IODINE.

Weigh out a small portion and expose it to the temperature at which it sublimes. All fixed impurities, such as black-lead, remain behind, and may be weighed.

To determine moisture, put a known quantity, say 30 grains, into a balanced capsule, add about eight times the weight of mercury, and weigh the whole along with a small agate capsule. Then rub the iodine and mercury carefully together with the pestle until the smell of iodine disappears, and the mass takes a reddish-brown colour. The dish is then placed in the water-bath, and after some time weighed. The loss of weight gives the quantity of water in the iodine.—*Bolley*.

Minute traces of iodine may be detected as follows:—Put into a test-tube a fragment of the peroxide of barium, add distilled water, pure hydrochloric acid, and starch-paste, when bubbles begin to appear on the surface, and lastly the suspected iodide. A rose, rose-blue, or blue shade will appear according to the quantity of iodine.

Chlorides, sulphurets, sulphites, and hyposulphites, do not interfere, only if the three latter are present, it is advisable to stir with a very fine glass rod.

Bromine interferes, as it furnishes a fine yellow colour with starch under the same circumstances; or—

The liquid or solution in question is concentrated in a porcelain capsule; absolutely pure carbonate of potash is added in slight excess, and boiled for some

time in order to decompose earthy or metallic salts present; cool and filter; evaporate the filtrate to dryness at a low temperature. The residue is powdered in the same capsule, and treated with alcohol of 40° Beaumè, to extract any iodides and bromides present. This alcoholic liquid is evaporated to dryness at a gentle heat, and if organic matter be present, this is destroyed by ignition at a low red heat. A few drops of dilute acetic acid are added, so as to be in slight excess. The liquid is again evaporated to dryness, avoiding a degree of heat that would decompose the acetate of potash. The residue is now dissolved in the smallest possible quantity of pure water, with two or three drops of a weak starch-mucilage recently prepared; then a small quantity of the test-liquor, a mixture of ten parts pure sulphuric acid at 66° Beaumè, and one part nitric acid at 25° Beaumè, is placed in a conical test-glass. The solution of the residue is poured very gently down the sides of the glass, so as to rest on the test-liquor without mixing with it. If iodides or bromides were present, there will quickly appear two zones in the saline solution, one of a clear topaz yellow, and another floating on it of a blue.—*Cantu.* Or—

Use benzine instead of starch-paste, effecting the liberation of the iodine by a few drops of nitrous acid, or by peroxide of barium and muriatic acid. Upon shaking the whole briskly, the benzine rises to the surface, carrying the iodine with it, with a splendid colour. *Bromine* and *chlorine* do not dissolve in benzine, and may thus be separated from iodine.

If the benzine solution of iodine is well washed in distilled water, and then shaken with a little nitrate of silver until decolorized, a yellow precipitate of iodide of silver is obtained, which is treated just like a precipitate of chloride of silver, and weighed. 100 parts of this precipitate represent 53.85 of iodine.

In this manner, very small traces of iodine may be quantitatively determined.

A current of hyponitrous gas may be passed into the solution in order to liberate the iodine, or the solution may be acidified with muriatic acid, and then a solution of the nitrite of potash added; starch-paste, or benzine, being of course present; or—

The iodine being liberated in any of the usual manners, bichloride of palladium is added; when, within about thirty-six hours, the brown iodide of palladium is deposited in flocks. This iodide may be washed by decantation, and if too small in amount to be collected, it may be mixed with a little moistened silica, collected, dried, and heated in a small glass tube over the lamp. The purple vapour of iodine is thus rendered perceptible.

The chief impurities found in commercial iodide of potassium are iodate, carbonate, and sulphate of potash, chlorides of potassium and sodium, sulphide of potassium, and organic matter containing sulphur. These impurities have in some cases amounted to upwards of 70 per cent.—*Pereira*.

Iodate of potash may be detected by adding a little tartaric or hydrochloric acid, when a deposit of iodine takes place. Carbonated and caustic alkalies may be

detected by the alkaline reaction of the sample on red litmus paper, and may be estimated, if required, by adding the test acid (II. § 1), as in alkalimetry. Carbonate and sulphate of potash also remain undissolved if the sample is treated with very strong alcohol. The alkaline chlorides if in large amount, are also left undissolved by alcohol. The chlorides may also be detected and estimated by precipitating the solution of the sample with nitrate of silver in excess, and adding an excess of ammonia. Iodide of silver remains insoluble whilst the chloride dissolves, and being again precipitated from the filtered liquid by the addition of nitric acid in excess, may be determined as in I. § 103.

The sulphuretted impurities may be detected by adding to the solution a little sulphuric acid and some fragments of clean zinc, and allowing the gas evolved to pass over some moist carbonate of lead, which will be blackened, or into a little of the solution of nitroprusside of potassium, which will turn purple if sulphur be present.

§ 13. IODIDE OF POTASSIUM.

1. Dissolve 10 grains bichromate of potash in one half ounce of cold water, and add a quarter of an ounce by measure of hydrochloric acid.

Make up an alkalimeter in the usual way, with 50 grains of the sample to be tested, dissolved in water, and the solution is added to that of the bichromate, until the chromic acid is completely decomposed. To find the point when the action is complete, spot a

white slab over with a mixture of pure protochloride of iron and sulphocyanide of potassium, very weak, and recently prepared. The spots should be large and thinly spread. By means of a glass rod, a small drop is brought in contact with one of these spots, touching the centre of the spot without any stirring. As long as a reddish tinge is produced, chromic acid still exists in the liquor, but when no dark shade is produced, the operation is finished. The number of degrees used is read off, and the per centage of iodine in the sample is found on dividing 5080 by this number, 10 grains of bichromate being equivalent to 25·4 of iodine.

Throughout the process, the liquid should be kept quite cool, the iodide should be added very slowly towards the end of the operation, and the liquid well stirred.—*Penny*. Or—

2. Dissolve 20 grains of the sample of iodide to be examined in 1000 grain measures of water. Dissolve meantime 8·28 grains of chloride of mercury (corrosive sublimate) in 1000 grain measures of water, and with it fill a pourette. Drop the mercurial solution carefully into the iodide, stirring frequently until the very faintest trace of a *permanent* red precipitate appears, whose permanence may be tested by vigorous stirring and setting aside for a few minutes. If the iodide be absolutely pure, the entire 100 degrees will barely suffice to produce the faintest precipitate; the number of degrees consumed showing the per centage of actual iodine in the sample.

§ 14. IODINE IN KELP AND SEA-WEEDS.

1. Exhaust a known weight with water; mix the several liquors, concentrate them and set them aside to crystallize. Decant off the mother-liquor, acidulate with muriatic acid, evaporate gently to dryness, adding a little carbonate of soda if the solution is acquiring a dark colour. Extract the residue in alcohol; decant off the alcoholic solution, mix it with water and heat to expel alcohol. Then drop it into the chrome solution as above.

Not less than 2 lbs. should be taken for a sample, and in case of "cut-weed" kelp only 5 grains of bichromate should be used in place of 10 grains.—*Penny*. Or—

2. Boil 500 grains of the sample, pulverized and well mixed with water, nearly neutralize the liquor and washings with nitric acid, and evaporate to a small bulk. The concentrated liquid is put in a platinum capsule and evaporated to dryness. The residue is fused, and kept in this state for a few minutes, when all the sulphur compounds are converted into sulphates without danger of decomposing the iodide. The fused mass is dissolved in water, the solution filtered if needful, and from 3 to 8 or 9 grains of nitrate of silver added. The mixture is heated and well shaken, when all the iodine is thrown down, and part of the chlorine. Nitric acid is now added in excess, and the mixture well washed by decantation. The last washing is decanted as completely as possible, and 250 grain measures of the strongest ammonia added, and

the whole well shaken. It is then digested for some hours, and the iodide of silver thrown on a small balanced filter and washed, first with liquid ammonia, then with hot water, dried and weighed. The weight of the iodide of silver, $+ \frac{1}{10}$ grain (dissolved by the ammonia) divided by 5, and multiplied by 0.54, gives the per centage of iodine in the sample.

In case of *kelp liquors* a cubic inch is nearly neutralized with sulphuric acid and boiled down to a small bulk; water is added and the solution is filtered to separate sulphur. Evaporate to dryness, fuse at a gentle heat, and introduce nitrate of potash in small successive portions until the melted mass is quite white, showing that all the sulphur has been oxidized. The analysis now proceeds as before. Care must be taken to use a sufficient quantity of ammonia. The amount of iodine obtained is multiplied by 288 to calculate the proportion for a gallon.

In determining the proportion of iodine in the various salts derived from kelp, 1000 grains are dried and heated with a little nitre to decompose sulphur compounds, the mass dissolved in water, and a large part of the salts crystallized out. The mother-liquor is treated with nitrate of silver as above.—*Wallace.*

§ 15. HYPOSULPHITE OF SODA.

Make a standard solution of perchloride of iron by dissolving a known weight of the best pianoforte wire in muriatic acid, passing chlorine gas in very large excess through the solution, and finally heating to expel the excess of acid. The liquid thus obtained

is made up with water to a known bulk. Dissolve 50 grains of the sample to be tested in water, and drop in the chloride of iron from a pourette as long as a violet colour is produced. 160·2 parts of actual perchloride of iron consumed represent 158·4 parts of dry hyposulphite of soda.

§ 16. ALUM.

This substance does not require a quantitative examination for the purposes of the dyer and colour-maker. If contaminated with iron it should be rejected, or only used after a thorough purification. To ascertain the presence of iron, add caustic potash in large excess to a boiling solution, and observe if a reddish-brown precipitate be formed. Mix a second portion of solution with nitric or nitro-muriatic acid, and boil until the excess of acid is driven off. Then divide the liquid in two parts; to the one add sulphocyanide of potassium, and to the other ferrocyanide of potassium (yellow prussiate). If iron be present the former will give a red colour, the latter a blue precipitate.

Ammonia-alum is distinguished from potash-alum by its greater solubility, and by giving off the odour of ammonia when heated with caustic potash.

Commercial *sulphate of alumina* may be tested for iron in the same manner as alum.

It may also be contaminated with sulphate of potash, which is detected by adding carbonate of ammonia in excess, filtering off the precipitate, concentrating the filtrate, evaporating to dryness, and heating to full

redness in a platinum capsule. If any residue appears alkali is present in the sample.

§ 17. COPPERAS (*Green Vitriol*).

Impure samples are to be totally rejected. Boil a portion of the sample in water with nitric acid, and add ammonia in excess. If the liquid has a blue tinge after the precipitate has subsided, copper is present.

Another portion of solution, likewise first prepared with nitric acid, is mixed with caustic potash in large excess, boiled and filtered. A solution of sal-ammoniac is then added. If, on standing, a white precipitate appears, the sample is contaminated with alumina, which is a very frequent impurity, arising from the presence of clay in the pyrites from which the copperas is prepared.

A third portion of solution is also boiled with nitric acid, mixed with a large excess of ammonia, and filtered. If HS , or hydrosulphate of ammonia produce therein a white precipitate insoluble in caustic ammonia and potash, zinc is present.

Lime is sometimes scattered over copperas to give it that brownish appearance which some purchasers prefer, not being aware that it is due to the conversion of a portion of the copperas into insoluble subsalts of peroxide of iron. If present it may be detected by adding slight excess of ammonia, which should be free from carbonic acid, allowing the precipitate to settle in a beaker, whose edges have been greased and covered with a well-fitting glass plate, to exclude

the air. A portion of the clear liquid is then decanted off, and some oxalic acid added, which, if lime be there, will give a white precipitate.

§ 18. SULPHATE OF COPPER (*Blue Vitriol*).

This substance is often offered for sale contaminated with iron, zinc, and magnesia.

To determine its value, see § 77.

Genuine sulphate of copper should contain, when in the crystalline state, 25.6 per cent. of metallic copper.

The impurities may be easily detected by the ordinary tests, after the copper has been removed by a current of HS.

§ 19. ARGOL (*Tartar or Cream of Tartar*).

1. Boil a weighed portion with dilute muriatic acid. Sand, if present, remains.

2. Take another portion (if the addition of the acid above has caused effervescence), and determine the carbonic acid (I. § 8, *b*). Every 11 parts of carbonic acid obtained represent 25 carbonate of lime present as an impurity.

3. The amount of lime may be determined in the solution No. 1 (I. § 10).

4. In the solution of a fresh portion, determine sulphuric acid, if present (II. § 6).

5. To ascertain whether the sulphuric acid found arises from alum, determine in another portion alumina, if present (I. § 14). If no alumina is found

the sulphuric acid represents an alkaline sulphate, generally soda.

In *tartaric acid* it is needful to ascertain the absence of sulphuric acid, of lime, or of tartrate of potash. If the latter impurity be found, carbonate of potash in proportional quantity remains, after igniting a sample in a platinum crucible.

If the sample, being free from potash or other base, reduces chloride of gold, and gives a white precipitate with nitrate of silver, oxalic acid is present. No saline matter should remain on the ignition of tartaric acid.

§ 20. OXALIC ACID ; OXALATES.

Heat a portion with sulphuric acid. If it turns brown or black, *organic impurities* are present.

If the sample be damp, *nitric acid* is probably present ; in which case the solution, boiled with a little sulphate of indigo, will discharge the blue colour of the latter.

Sulphates, if present, are detected and estimated in the ordinary manner.

Pure oxalic acid should leave no residue if heated to redness in a platinum capsule.

Binoxalate of potash (salt of sorrel or sal-acetos), is examined in the same manner. If pure, it leaves, when heated to redness, an equivalent quantity of carbonate of potash. It is sometimes improperly called "salt of lemons"—a dangerous error.

§ 21. CITRIC ACID AND LIME JUICE.

Crystalline citric acid is generally pure. In sam-

ples of lime juice evaporate a portion to dryness, and ignite to detect alkaline salts. Test for sulphuric acid, and determine its amount, if present, in the usual manner. If lime water, added to a cold dilute solution in sufficient quantity to render the liquid slightly alkaline, give an *immediate* white precipitate, oxalic acid is present. If solution of gypsum produce a precipitate, either oxalic or para-tartaric acid is present. The presence of tartaric and para-tartaric acids may also be shown by adding a solution of the nitrate or sulphate of potash, which, in that case, will give a white crystalline precipitate.

§ 22. SUCCINIC ACID.

Succinic acid is often contaminated to a greater or less extent with ammoniacal and alkaline salts, with tartaric acid and empyreumatic oil. It should leave no residue when ignited on a slip of platinum foil. A carbonaceous residue shows the presence of tartaric acid, a saline residue that of some salt of potash or soda. If the odour of ammonia is given off when the substance is warmed with a solution of potash, sal-ammoniac, or some other ammoniacal salt is present. The acid should be quite white, and perfectly soluble in alcohol. If added along with an excess of ammonia to the solution of a per-salt of iron, the iron should be precipitated.

Succinate of ammonia should likewise be perfectly colourless, and volatile without residue at a red heat. To detect the presence of sal-ammoniac add a little pure

carbonate of soda, evaporate down to dryness, ignite the residue on a platinum capsule, take up the residue in a few drops of water, acidulate with nitric acid, and test for chlorine with nitrate of silver in the usual manner.

§ 23. HYDROFLUORIC ACID.

This acid has now become an article of commerce, and is offered for sale at about one shilling per pound.

Its saturating power or real strength may be estimated by means of the standard solution of pure carbonate of soda (II. § 1). Each grain of pure carbonate of soda consumed indicates 0.35 grain of actual hydrofluoric acid, provided that no other acid is present.

If other acids are present as impurities, the liquid may be carefully neutralized with baryta water, and the precipitate filtered and washed. If hydrochloric acid be present, the chlorine found in the filtrate as chloride of barium may be determined (II. § 4).

The precipitate is dried, weighed, and treated with hydrochloric acid. Fluoride of barium dissolves, and sulphate of baryta, if sulphuric acid be present, is left untouched along with perhaps some silica. The latter might be removed by digestion in some free hydrofluoric acid.

Silica in hydrofluoric acid may be detected by adding the fluoride of potassium, which throws down the double fluoride of silicium and potassium as a

gelatinous precipitate. Decant off the liquid and boil the precipitate in carbonate of potash. Silica is deposited, and may be washed by decantation, dried, ignited, and weighed.

Alkaline or earthy salts, or other fixed matter (except silica and boracic acid), will remain behind, if a portion of the acid be evaporated away—an operation which must be performed under a chimney with a good draught, as the fumes are highly pernicious. All these operations must be performed in vessels of platinum.

Note.—The fluorides of manganese and chrome are also volatile, but these substances are not likely to contaminate hydrofluoric acid.

If galena is found in fluor-spar, the hydrofluoric acid prepared from it is turbid with sulphur.

§ 24. HYDROCYANIC ACID (*Prussic Acid*).

a. To determine the amount of actual hydrocyanic acid in a sample, mix some nitrate of silver with a little ammonia, so that the resulting clear liquid may be rather acid than alkaline, and pour it into a weighed portion of the sample as long as any precipitate of cyanide of silver is produced. The precipitate is collected on a small filter previously dried at 212° Fahr., and weighed, and the precipitate and filter are washed, dried at 212° Fahr., and weighed. 133.9 parts cyanide of silver represent 27 of anhydrous hydrocyanic acid.—*Duflas*.

b. Add to the liquid containing prussic acid some caustic potash or soda until it colours red litmus paper decidedly blue. Dissolve some pure nitrate of silver,

accurately weighed, in a known volume of water, so as to form a standard solution. This is now carefully dropped from a pourette into the hydrocyanic liquid, with frequent stirring, until a slight turbidity begins to appear. The number of degrees of the silver solution consumed shows the quantity of real prussic acid present in the sample, since 170 parts of pure crystalline nitrate of silver represent 54 parts of anhydrous prussic acid. This method is applicable to bitter almond water and laurel water. Turbid bitter almond water must first be mixed with three or four times its volume of water to render it clear.—*Liebig*.

If prussic acid is contaminated with any of the stronger acids, litmus paper reddened by immersion in the acid, retains its red colour after prolonged exposure to the air. The colourless solution of the compound of cyanide of mercury with iodide of potassium throws down a scarlet precipitate with prussic acid, contaminated with one of the stronger acids. Sulphuric acid occasions a turbidity with chloride of barium, insoluble in hydrochloric acid.

To detect hydrochloric acid the sample is mixed with pure ammonia, and evaporated on the water-bath. Sal-ammoniac will remain if hydrochloric acid was present. Or, if a small quantity of pure borax is dissolved in the acid, and the liquid evaporated to perfect dryness in the water-bath, the aqueous solution of the residue gives a precipitate with nitrate of silver, if the original acid contained hydrochloric acid. Or, if excess of nitrate of silver is added, the precipitate

formed does not completely redissolve in hot strong nitric acid, if hydrochloric acid was present.

For detecting traces of *nitric acid*, see II. § 5 ; also II. § 3, 2.

If SH is present the liquid gives a brown precipitate with a solution of cyanide of mercury.

If *ammonia* is present the sample treated with strong potash, in the cold, gives off ammonia, recognizable by the white fumes which it forms with a glass rod dipped in hydrochloric acid.

Traces of *lead* may be detected by adding sulphuric acid and concentrating, or by passing a current of SH through the liquid, which will give a blackish brown precipitate.

Traces of *mercury* are also detected by SH (see also the tests for mercury, II. § 160).

Formic acid is detected by dissolving some red oxide of mercury in the liquid, and heating the clear solution when the mercury is reduced, producing a grey cloud, and a deposit of globules of mercury. Or, if the acid thus contaminated be digested with carbonate of lead, on evaporating the filtrate, needles of formiate of lead are obtained.

If hydro-sulphocyanic acid is present, a few drops of persulphate or perchloride of iron produce a deep red colour.

If a residue appear, after evaporating a portion of the sample to dryness on the water-bath, it may consist of phosphoric or sulphuric acid, sulphate of potash, bitartrate of potash, oxide of lead, cyanide of mercury, and prussian blue.

§ 25. CYANIDE OF POTASSIUM.

Prepare a standard solution of the ammonio-sulphate of copper as follows:—Take dry pure crystalline sulphate of copper, made by bruising the crystals in a mortar, and drying them between folds of blotting paper. Of this dissolve 390.62 grains in water and make up to 2000 grain measures. Every 100 grain measures of this correspond to 5 grains metallic copper.

Take 100 grains of each sample of cyanide to be tested, dissolve in water in a beaker placed upon a sheet of white paper, add an excess of ammonia, and then pour in the copper solution from a pourette, stirring diligently till a very faint blue tinge begins to appear in the liquid. The number of degrees consumed in each case indicates the relative value.

If it be desired to find the actual amount of pure cyanide in a commercial sample, take pure hydrocyanic acid, determine its strength (II. § 24), then supersaturate with ammonia such a quantity of this acid as shall be equivalent to 100 grains of pure cyanide of potassium, and test it with the copper solution. The exact value of this solution being thus known, the amount of actual cyanide contained in each sample may be accurately found.—*Herapath*. Or—

Proceed as in II. § 24, *b*. (Liebig's method), the preliminary addition of caustic alkali being of course omitted.

The principal impurities in commercial cyanide of potassium are—*Carbonate of potash*; this remains behind when the cyanide is dissolved in hot alcohol

of 36° Beaumè, and may be recognized as such by its usual properties. On agitating impure cyanide with cold alcohol of 78 per cent., the carbonate of potash dissolves and forms a fluid layer at the bottom. *Sulphide of potassium*, if present, causes the solution to give with salts of lead, not a white, but a dirty brownish precipitate. If an excess of dilute sulphuric acid be added; the gas given off darkens paper coated with moist carbonate of lead. *Alkaline sulphates*, after supersaturation with muriatic acid, cause a white precipitate with chloride of barium. *Alkaline chlorides*; the sample ignited with twice its weight of nitre and ten times its weight of carbonate of soda (both of course perfectly free from chlorine), then dissolved in water, and mixed with excess of nitric acid, gives a precipitate with nitrate of silver, if chlorides were present. *Silicate of potash* leaves a residue of silica, after addition of excess of muriatic acid, evaporation to dryness, and resolution in water. *Ferrocyanide of potassium*; the solution mixed with a small quantity of perchloride of iron perfectly free from any trace of protosalts of iron, and then with muriatic acid, gives a precipitate of prussian blue, if any trace of ferrocyanide is present. Or, the precipitate given by sulphate of copper turns not white, but reddish, on the addition of muriatic acid. *Sulphocyanide of potassium*; the solution mixed with an excess of muriatic acid turns perchloride of iron red. *Cyanate of potash*; if that portion of the sample which is soluble in alcohol of 36° Beaumè, gives off carbonic acid when mixed with dilute acids, and precipitates carbonate of baryta from salts of

baryta, the presence of cyanate of potash may be inferred. *Formiate of potash*; the sample blackens on ignition. Or, on passing carbonic acid gas through the solution till all the hydrocyanic acid is expelled, exhausting the evaporated mass with alcohol, evaporating to dryness, and distilling the dry residue with dilute sulphuric acid, the formic acid distils over, and may be recognized by its power of reducing salts of mercury and silver.

§ 26. FERROCYANIDE AND FERRID-CYANIDE OF POTASSIUM
(*Yellow and Red Prussiates*).

Prepare a standard solution by dissolving 200 grains of ferrocyanide of potassium (in perfectly pure dry crystals) in water, and make up the solution to 10,000 grain measures.

Prepare also a solution of permanganate of potash, of such strength that the contents of an ordinary pourette (1000 grain measures) may be nearly all consumed by 100 grain measures of the former solution.

The value of the permanganate liquor is first determined by measuring off with a small pipette 100 grain measures of the ferrocyanide liquor, pouring it into a beaker, diluting with about 25 times its bulk of water, and acidulating with muriatic acid. The beaker is set upon a sheet of white paper and the permanganate dropped into it, constantly stirring until an orange, or reddish-yellow tint appearing in the liquid, shows that the reaction is complete.

As the solution of permanganate is liable to change,

its value should be determined before every fresh series of experiments.

To test a sample of yellow prussiate, dissolve 50 grains in 2500 grain-measures of water, and 100 grain-measures of this are taken and tested as above.

The calculation of results is very simple; 100 grain measures of the standard ferrocyanide solution contain two grains of pure ferrocyanide. Suppose that 90 degrees of the permanganate have been required to saturate 100 grain measures of the standard ferrocyanide, and that only 70 have been consumed in testing the same amount of a commercial sample, then $90 : 70 = 2$ (number of grains of pure prussiate contained in 100 grain measures of the standard solution) : x (number of grains of pure prussiate contained in 100 grain measures of the solution of the sample tested).

For testing samples of red prussiate, the same quantity is taken, dissolved in the same bulk of water, and 100 grain measures of the sample taken as before. To this portion are added 50 to 80 grain measures of concentrated solution of potash, and the whole is boiled in a porcelain basin, with six or seven grains of oxide of lead in fine powder. The lead turns brown and the liquid pale yellow, leaving no doubt as to the change having taken place. Dilute with water, filter, wash, and add water, till the whole amounts to 2500 grain measures. Acidulate with muriatic acid; and, disregarding the white turbidity produced, add the permanganate as before,

until the change of colour takes place, the turbidity disappearing at the same time.

The same calculation as before gives the amount of yellow prussiate found in the fluid after reduction, which is of course equivalent to the amount of red prussiate previous to that process; 52.81 of the former representing 41.17 of the latter.

If it be required to analyse a mixture of the two prussiates, the amount of ferrocyanide is first determined as above, and a similar quantity tested again after treatment with potash and oxide of lead. The amount of red prussiate is found by multiplying the difference of the two determinations by 0.7795.

§ 27. CHROMATE AND BICHROMATE OF POTASH.

These salts frequently contain, the former, alkaline carbonates; the latter, sulphates. In the first case, dilute sulphuric acid causes an effervescence; in the latter, the solution mixed with muriatic acid gives a white insoluble precipitate with dilute chloride of barium.

To ascertain the quantity of real chromic acid present in a sample of either, dissolve 100 grains in two ounces of water, with half an ounce of muriatic acid, and add a known excess of a solution of protochloride of tin whose strength has been determined as in II. § 35. The excess of tin remaining in the state of protochloride over and above the amount converted into a per-salt by the chromic acid of the sample, is then again determined as in II. § 35. Dry also a weighed portion, and note the loss of moisture.

Black mordant.—This is simply bichromate ground to powder, and mixed with common salt or any other cheap article, to mask its appearance. It actually finds purchasers in the west of Yorkshire at a higher price than the best quality of genuine bichromate! It is examined as bichromate.

§ 28. CHLORATE OF POTASH.

Dissolve in water and add nitrate of silver; if any alkaline chloride be present, a white precipitate will be produced. The amount of this impurity is easily ascertained (I. § 1, *b*). No nitric acid should be added.

§ 29. ACETATE OF SODA.

Dry a portion carefully, and note the amount of moisture lost. Dissolve in water, filter off, and weigh any insoluble residue. The filtered liquid is divided into two equal portions by means of a pourette. In one of these, chlorine is determined, if present (I. § 1, *b*, or II. § 4), and accounted for as chloride of sodium, 35.4 parts of chlorine representing 58.6 parts of chloride of sodium or common salt. In the other portion determine sulphuric acid if present (II. § 6), and account for it as sulphate of soda; 40 parts of sulphuric acid representing 71.2 of dry sulphate of soda. Lime may be detected by adding oxalate of ammonia, when it will fall as a white precipitate. If iron is present, hydrosulphate of ammonia will give a black precipitate.

§ 30. ACETATE OF LEAD (*Sugar of Lead*).

Sugar of lead should be entirely soluble in water. Any insoluble matter may consist of carbonate of lead, sulphate of lead, sand, and lime. If the addition of ammonia in excess gives a blue tinge, copper is present. A brown tint, very common in the inferior qualities of sugar of lead, may arise from the empyreumatic matter accompanying crude pyroligneous acid; but it may also be due to iron. To determine this, add red prussiate of potash, and note if a blue precipitate is formed; or, take a small portion and boil with nitro-muriatic acid to peroxidize, expel excess of acid by evaporation, add a little water, and then test one portion with yellow prussiate and another with sulphocyanide of potassium. If the former give a blue precipitate, and the latter a red colour, iron is present. To detect lime, remove the lead from a portion of the solution, by a current of SH, decant off the clear liquid, heat to expel excess of SH, add a little ammonia, and then oxalate of ammonia. A white precipitate shows lime.

§ 31. ACETATE OF LIME.

Dissolve in water to which a drop or two of alcohol has been added. Sulphate and carbonate of lime will remain undissolved and may be filtered off. Test the clear liquid for sulphuric acid, and for chlorine, which, if present, may be determined as in II. §§ 4 and 6. The sulphuric acid will belong to sulphate of soda; the chlorine to chloride of sodium, or of calcium. If

it be desired to determine which of these two impurities is present, make a fresh solution of 100 grains in dilute alcohol, and evaporate the filtered liquid to dryness. Ignite very cautiously in a platinum capsule, raising the heat very gradually. In one half of the residue, which must, of course, be accurately weighed, determine *lime* (I. § 10), and in the other carbonic acid (I. § 8, *a* or *b*). If there be found more lime than will combine with the carbonic acid, *i. e.*, more than twenty-eight parts of the former to twenty-two of the latter, the excess of the lime must have been in combination with chlorine.

§ 32. ACETATE OF ALUMINA (*Red Liquor*),

If pure, gives off all its acid below redness, alumina alone remaining. It often contains sulphates of alkali and of alumina, from excess of alum (determine sulphuric acid), chlorides of alkali (determine chlorine), and traces of iron. The latter is a very objectionable impurity.

To detect it, add red prussiate, which will give a blue precipitate if iron be present; or, peroxidize by heating with nitric acid or *aqua regia*, and test with yellow prussiate, or sulphocyanide of potassium, as in II. § 30. To add to the original solution hydrosulphate of ammonia would be an unsafe test, since traces of lead may be present which would give a black precipitate with that reagent, and might thus be mistaken for iron. To determine iron, if required, proceed as in I. § 23, by which the respective amounts of iron, alkali, and alumina may be ascertained; or,

ignite the sample, dissolve the residue in muriatic acid, precipitate with ammonia, filter off, and wash the precipitate, and separate alumina from iron, as in I. § 14. The filtrate evaporated to dryness after the addition of sulphuric acid in slight excess, and gently ignited, leaves the alkali as sulphate.

Traces of lead are not injurious.

§ 33. ACETATE OF IRON (*Iron Liquor*).

Determine sulphuric acid and chlorine, if present. The former proves the presence of green vitriol, the latter of common salt. Add to a portion muriatic acid and chlorine water, or a little nitric acid, with the aid of heat to peroxidize the iron; which may then be determined by the addition of ammonia (I. § 15). The filtrate is then evaporated to dryness and heated. No fixed matter should remain, except a trace of carbon from the tarry matters found in raw pyroligneous acid.

§ 34. NITRATE OF IRON, PERSULPHATE OF IRON, PERCHLORIDE OF IRON, SULPHO-NITRATE OF IRON.

Determine the amount of iron as in I. § 15. If any fixed matter remain after evaporation to dryness and ignition, examine for earthy and alkaline chlorides, nitrates, and sulphates. If alumina be present as in samples prepared from copperas, see I. § 14.

To detect the presence of unperoxidized iron, add cautiously dilute carbonate of soda from a dropping tube. The peroxide falls down first, and any protoxide which may be present appears as a greenish

cloud, on the addition of more carbonate of soda. The red prussiate of potash gives doubtful results, if nitric oxide be present.

§ 35. TIN CRYSTALS (*Protochloride of Tin*).

Take an average sample, weigh out 100 grains, and dissolve in two ounces of water, adding half an ounce pure hydrochloric acid, at a gentle heat.

Dissolve 83.2 grains of pure, dry bichromate of potash in two ounces of warm water. Put the solution into a common pourette, which is filled up with water, thoroughly mixed by closing the apertures with the fingers, and inverting two or three times. Each degree of the pourette represents one grain of metallic tin. Add now to the tin solution a little iodide of potassium and starch paste. Drop in the bichrome solution, stirring frequently, until a blue tinge appears in the liquid. The number of degrees of the pourette consumed, shows how many grains of actual tin are contained in the sample.

This method is not applicable if protochloride of iron is present. In that case, prepare a solution of perchloride of iron, and ascertain its value by dissolving a known weight of tin in hydrochloric acid, adding a little sulphocyanide of potassium, and pouring in the chloride of iron with a pourette, until all the tin is converted into perchloride; a change shown by the appearance of a red colour. During this process, the tin liquid should be kept boiling hot, and be often stirred. The value of the iron solution being thus known, it is applied in the same manner to the sample,

and the per centage of tin is known from the number of degrees consumed.

Protochloride of tin present in perchloride may be detected by adding a little chloride of mercury in solution, which will give a white precipitate that quickly turns black.

To detect perchloride in protochloride, dissolve a portion of the sample in water (not so dilute as to occasion any milkiness), and add a solution of sulphate of soda in excess, a precipitate will appear at once, or in some minutes. The solutions should be mixed in a small corked or stoppered flask, to exclude air. Any excess of acid should be carefully neutralized with ammonia.

§ 36. SINGLE AND DOUBLE MURIATE OF TIN

(Dilute and concentrated solutions of Protochloride of Tin).

Pour 500 grains of the sample into a beaker adding a little iodide of potassium and starch paste, as before. Make a solution of pure bichromate of half the strength indicated above, and with it make up a pourette in the same manner; one degree of this will indicate half a grain of metallic tin. Drop in the chrome to the tin, and note the degrees consumed.

The method with iron may also be employed. The muriates of tin are often mixed with zinc. They are commonly supposed to contain respectively 12 and 25 per cent. of tin; amounts which are impossible at their usual prices.

Traces of perchloride may be detected as in II. § 35.

§ 37. OXYMURIATE (BICHLORIDE) OF TIN.

Add to the solution pure metallic zinc. The tin is thrown down in the metallic state, and is redissolved in hydrochloric acid and determined as above. Sulphuric acid, which is often added to get up the specific gravity of these solutions, is determined as in II. § 6.

Traces of protochloride are detected by chloride of mercury, as in II. § 35.

§ 38. RED SPIRIT, YELLOW SPIRIT, BARWOOD SPIRIT, PLUM SPIRIT (*Mixtures of Protochloride and Perchloride of Tin*).

Determine tin present as protochloride, as in II. §§ 35 and 36. Then determine total tin as in II. § 37. The former amount subtracted from the latter, shows the quantity of tin present as perchloride. Muriatic and sulphuric acids are determined, if requisite, in the usual manner. Iron is detected by boiling with nitric acid to peroxidize, expelling free acid by evaporation, and adding sulphocyanide of potassium, when a red tinge will appear. To detect zinc, expel excess of acid, and pass a current of HS for a long time through the liquid. Filter off the precipitate, and mix the filtrate with hydrosulphate of ammonia. A white precipitate, insoluble in caustic potash, shows the presence of zinc.

§ 39. OXALATE OF TIN.

The mordant sold under the name oxalate of tin,

is generally a chloride of tin, to which some oxalic acid or oxalate of potash has been added. Free muriatic, and sometimes sulphuric acid are also present.

Tin is determined as in II. §§ 35, 36, 37, noting whether it exists as proto- or per-salt, and how much of each.

To detect and determine the oxalic acid, partially neutralize it with ammonia, leaving it still an acid reaction. Then pass a current of SH through the solution for a very long time. Filter off the precipitate (sulphide of tin), and heat gently the filtrate to expel excess of gas. Then neutralize exactly with pure carbonate of soda, evaporate to dryness, and ignite. The residue, which consists of chloride of sodium, sulphate of soda (if sulphuric acid was present), and carbonate of soda derived from the decomposition of the oxalate of soda, is dissolved in water, and the solution divided into three known parts. In one determine sulphuric acid (II. § 6); in another chlorine (II. § 4); and in the third carbonic acid (I. § 8, *b*). Every 22 parts of carbonic acid found, represent 45 parts of dry oxalic acid, or 63 parts in its ordinary crystalline state.

If no sulphuric acid be present, the liquid, after removal of the tin, may be neutralized with pure ammonia and chloride of calcium added. The oxalic acid falls as oxalate of lime. See I. § 10.

The muriatic acid is then determined, if required, in a separate portion.

An oxalate of protoxide of tin cannot exist in solu-

tion, except in presence of a considerable excess of muriatic acid.

Much of the so-called "oxalate of tin" contains a mere trace of oxalic acid, or even none at all.

§ 40. PINK SALT (*Double Chloride of Tin and Ammonium*).

The sample is dissolved in excess of water and boiled, when the whole of the tin is deposited as stannic oxide in white flakes, which are filtered off, washed, ignited, and weighed; 74.82 parts of stannic oxide represent 58.82 parts of metallic tin.

§ 41. STANNITES OF ALKALI.

Add metallic zinc to the solution. Wash the precipitate of metallic tin, dissolve in hydrochloric acid, and determine as in II. § 22.

Stannates of Alkali.—Dissolve the sample in the smallest possible quantity of water, add an excess of concentrated nitric acid, and boil until the stannic oxide which is at first thrown down in the soluble state, becomes insoluble. This change is indicated by the disengagement of ruddy fumes. The solution may then be diluted with water, filtered, the precipitate washed, dried, ignited, and weighed as stannic oxide.

§ 42. PHOSPHATE OF SODA (*Commercial*).

Dissolve in water; phosphate and sulphate of lime may remain undissolved. Examine the solution for sulphuric acid, and chlorine which may be present as sulphate of soda and common salt. If a drop of

muriatic acid causes effervescence, carbonate of soda is present, more having been used than the phosphoric acid could take up.

Phosphoric Acid may contain a variety of impurities. If phosphorous acid is present a solution of chloride of mercury gives a white precipitate, which turns black. If nitric acid is present, it may be detected by the tests given (II. § 3, 2). Sulphuric acid gives with chloride of barium a white precipitate, insoluble in muriatic acid. Ammonia may be detected by heating with excess of lime. Lime and magnesia are precipitated by ammonia, especially with the addition of a little oxalate of ammonia. Arsenic is precipitated yellow by a current of SH. Lead and copper are thrown down by SH, as deep, black-brown precipitates, from the diluted liquid. Iron gives a red coloration with sulphocyanide of potassium.

Phosphorus rarely contains any other impurity beside a little phosphoric oxide, which gives it a reddish colour; and traces of arsenic, which may be detected as in II. § 43.

The latter impurity only affects its value for scientific or medical purposes.

§ 43. ARSENIC (*Arsenious and Arsenic Acids*).

If the arsenic is present as arsenious acid alone, mix the substance with a weighed excess of bichromate of potash along with oxalate of soda. Place the mixture in the inner tube of the apparatus I. § 8, *b*, and proceed as there directed.

Now, three equivalents or 297 parts of arsenious

acid, require for their oxidation to arsenic acid, 203.228 of chromic acid, which amount is contained in 297.516 bichromate of potash. But 297.516 of bichromate, in contact with oxalate of soda and free sulphuric acid, would evolve 264 parts of carbonic acid; so that, for every 297 parts of arsenious acid present, 264 parts less of carbonic acid will be generated than the same amount of bichromate, oxalate, and sulphuric acid would yield in the absence of the arsenious acid.

If the arsenic be present as arsenic acid, the latter is first reduced to arsenious acid by a current of sulphurous acid gas, and then treated as above.

If arsenious and arsenic acid are jointly present, the former is first determined in one portion, and another sample is then treated with sulphurous acid and determined as above. The amount of arsenic acid is calculated from the difference of the two determinations. If chlorine be present, oxide of mercury must be added.—*Vohl*.

The chief impurity in arsenious acid is antimony. If this is the case, the hydrochloric acid solution of the sample is precipitated, of an orange instead of yellow by a current of SH. It is sometimes adulterated with other heavy white mineral substances, such as sulphate and carbonate of baryta, sulphate of lead, &c. All such contaminations may be detected by igniting a small portion in a capsule (under a good draught), when, the arsenious acid being volatile sublimes, leaving the impurities.

The best tests for arsenious acid are SH, which

gives a yellow precipitate in the solution, a little muriatic acid having been previously added. Ammonia-nitrate of silver causes also a yellow precipitate, soluble in nitric acid and in ammonia.

Where, from the colour of the liquid or the presence of other substances, these tests are inapplicable, Reinsch's test may be used. The process is as follows:—The substance in question is dissolved in muriatic acid, and diluted with water. If a liquid, it is mixed with a rather large amount of muriatic acid. A slip of clean, bright copper foil, free from arsenic, is introduced, and the whole boiled in a flask for eight to ten minutes. Chlorate of potash must not be present. If arsenic is present, it will form a steel-gray coating on the copper. The copper may then be withdrawn from the liquid, allowed to dry, and being introduced into a very small and narrow test-tube, may be sublimed off the copper, and thrown upon the interior of the glass by the heat of a gas flame. Here it may be dissolved, by heating in a little aqua-regia, and submitted to the tests for arsenic acid.

Marsh's apparatus is, in fact, simply a flask fitted up for gas-generating. It is charged with some fragments of pure zinc, and dilute sulphuric acid. The current of gas is dried by passing through a chloride of calcium tube, and is then conducted through a narrow tube of hard German glass, free from lead, which is connected with a Liebig's bulb-tube, half filled with solution of nitrate of silver. As soon as the common air is expelled from the apparatus, the middle of the narrow hard glass tube is heated

with a gas-flame. If no steel-grey mirror-like coating appears in the tube a little beyond the heated spot, and no deposit of reduced silver appears in the bulb-tube, the zinc and sulphuric acid may be judged to be pure, and the solution of the suspected substance is poured into the flask by the tube-funnel, which, of course, passes below the level of the liquid in the flask. If arsenic is present, a metallic mirror will coat the inside of the tube a little beyond the heated spot, and at the same time, some silver in the bulb-tube will be reduced. When the action is over, the hard glass tube is cut with a file, and the part where the mirror lies, if large enough, divided into a few portions. If one of these is put into a strong solution of chloride of soda, it should dissolve off, leaving the glass clear. If another is placed in a small test-tube and heated, it should readily sublime, or admit of being chased up and down the tube. Another fragment may be introduced into a narrow glass tube open at both ends, and heated whilst held in a slanting position. The metallic crust should change into a white powdery sublimate, which may also be readily chased up and down the tube by heat; and which, when examined with a lens, appears composed of minute crystals.

It may then be dissolved in a little muriatic acid, and the solution, when mixed with water and treated with a current of SH , should give a yellow precipitate. By these tests the metallic deposit is proved to be arsenic, and not antimony—the only body with which it is liable to be confounded.

Arsenic acid gives also a yellow precipitate with SH, but it does not form for some time if the solution be very dilute. With the ammonia-nitrate of silver, arsenic acid gives a red precipitate. Reinsch's test is also less rapid and less delicate for arsenic acid than for arsenious acid.

§ 44. ARSENITE AND ARSENIATE OF SODA.

These salts should be perfectly soluble in water, should not effervesce if their solutions are mixed with dilute muriatic acid—a proof of the presence of carbonate of soda. They should likewise be free from sulphates, chlorides, and nitrates or nitrites; the two latter of which are often found in arseniate of soda, prepared by deflagrating arsenious acid with nitrate of soda. For the detection of nitrates and nitrites, see II. § 5, 3.

§ 45. MANGANATES AND PERMANGANATES OF POTASH AND SODA.

These substances are now become articles of commerce, and are used especially as disinfecting agents for destroying organic impurities in sewage, rivers, &c.

The impurities which may be present in a sample are sand and undissolved peroxide and oxide of manganese, silicate, and aluminate of alkali; besides alkaline nitrates, nitrites, sulphates, and chlorides.

To ascertain how much real permanganate or manganate is present in a sample, prepare a test-liquid, by dissolving ten grains of pure tin in pure hydro-

chloric acid, and add water, so as to make it up to a known volume.

To find the value of this liquid, dissolve some pure crystalline permanganate of potash in about three or four times its weight of water; noting, of course, the exact weights employed. Into the red solution thus obtained, drop the tin-liquid from a pourette, until only a very faint rose tint remains, which another drop would destroy. The value of the tin solution being thus found, the samples to be examined are dissolved each in about four parts of water; and if manganates, are converted into permanganate, by carefully adding nitric acid until the liquid takes a fine crimson-violet tint.

The tin solution is then added from a pourette as above, until only a faint pink colour remains; when, from the number of degrees consumed, the amount of real permanganic acid in each sample will appear.

The manganate of potash contains 52.4 per cent. of manganic acid, the permanganate, 70.35 per cent. of permanganic acid.

The corresponding soda salts contain respectively 62.5 and 78.2 of manganic and permanganic acids, and are of course more valuable.

The impurities present do not interfere further than by diminishing the per centage of available manganate or permanganate, and require no special examination.

§ 46. SULPHATE OF MANGANESE.

The only impurity of consequence is iron. If the sample is free from this contamination, it will give,

with the yellow prussiate of potash, a white precipitate, which on long standing, shows no tinge of blue, and with hydrosulphate of ammonia, a pure flesh-coloured precipitate without any dusky or blackish appearance.

The chloride (or nitrate) of manganese is liable to the same impurity, and is examined in the same manner.

§ 47. NITRATE OF COPPER.

This salt is sold in a liquid state to dyers, and is largely mixed with articles calculated to increase the specific gravity of the solution. The best method of determining its value is to add ammonia in excess, until the precipitate first formed redissolves, and then proceed as in II. § 152.

§ 48. NITRATE OF LEAD.

The nitrate of lead should be crystalline, without any tendency to grow damp, and perfectly soluble. The precipitate caused in the solution by yellow prussiate of potash, should be white, without any shade of blue (iron), or chestnut (copper). If the lead is entirely removed, by passing a current of SH through the liquid for a considerable time, the filtered solution, evaporated to dryness and ignited, should leave no residue.

§ 49. ALKALINE PLUMBITES.

In a portion of the liquid determine carbonic acid (I. § 8, b). Heat another portion with excess of nitric acid, evaporate down to remove superfluous

acid, pass a current of SH through the solution, filter off the precipitate of sulphide of lead, and add hydrosulphate of ammonia. No precipitate should appear.

§ 50. SULPHATE OF ZINC (*White Vitriol*).

If ammonia added in excess gives a blue colour to the liquid, copper is present. If the hydrosulphate of ammonia gives a pure white precipitate, the sample may be judged free from iron; but if the precipitate be greyish, or still more black, that metal is present.

Take another portion of the sample, and boil with a large excess of pure caustic potash. Nothing should remain undissolved. If a current of SH is passed through the clear liquid until all the zinc is precipitated as sulphuret, and this be allowed to subside, the clear liquid decanted off should give no precipitate when mixed with excess of sal-ammoniac.

§ 51. SULPHATE OF MAGNESIA (*Epsom Salt*).

If a portion be dissolved in water, and the cold solution be well shaken up with an excess of carbonate of baryta, and the liquid then becomes strongly alkaline, *sulphate of soda* (a very common impurity) is present. Or, precipitate the solution with baryta water, and then add carbonate of ammonia in excess. Filter, evaporate the filtrate to dryness, and ignite. If sulphate of soda be present, carbonate of soda will remain. If *chlorides of magnesium or sodium* be present, add some nitric acid, and a very dilute solution of nitrate of silver will give a white precipitate. If hydrosulphate of ammonia gives a black precipitate,

iron is present. If excess of ammonia gives a blue colour, or a current of SH a brown precipitate, *copper* is present. To detect *manganese*, add bicarbonate of soda, and pass a current of chlorine gas through the solution, when a brown precipitate will appear.

§ 52. CALCINED MAGNESIA.

Treat a portion of the sample with water, filter off the liquid, and concentrate. Divide into two portions, add to the one muriatic acid and chloride of barium. If a white precipitate appears, *alkaline sulphates* are present. Mix the other with pure nitric acid, and add nitrate of silver. A white precipitate betrays the existence of *alkaline chlorides*. To another portion of the sample add hydrochloric acid; in which it should dissolve without effervescence (which shows the presence of *carbonate of magnesia*) and without residue. Any portion left undissolved is *silica*. Neutralize a part of the muriatic solution with ammonia, add sal-ammoniac and some oxalic acid. A turbidity or precipitate shows the presence of lime. It should give no precipitate with SH, nor with hydrosulphate of ammonia well saturated with sulphur.

§ 53. CARBONATE OF MAGNESIA (*Magnesia alba*).

This substance is examined as in II. § 52. It should be insoluble in water, perfectly soluble in muriatic acid, and should be carefully examined for lime, which is often present to a great extent in the form of chalk.

§ 54. OXIDE OF ZINC (*White Zinc, Philosopher's Wool*).

Treat a part of the sample with water; nothing should be dissolved. Test the liquid for sulphuric acid and chlorine, which, if found, represent *sulphate and chloride of alkali*. Digest another portion in dilute hydrochloric acid; it should dissolve completely without effervescence (*carbonates of zinc, lime, magnesia, and baryta*), and without evolution of hydrogen gas (*metallic zinc*). Any matter left undissolved, may consist of *sulphate of baryta, china clay, and silica*. Add to the muriatic acid solution excess of well-sulphured hydrosulphate of ammonia. Filter off the precipitate of sulphuret of zinc, concentrate the clear liquid, and divide into three portions, *a, b, and c*. To *a* add a concentrated solution of gypsum. A white precipitate shows the presence of *carbonate of baryta* in the sample. To *b* add a little ammonia, so as to nearly or quite neutralize it, and then oxalic acid. A white precipitate shows *carbonate of lime*. If neither of these be present, add to *c* ammonia, so as to neutralize it, and then some phosphate of soda. If *carbonate of magnesia* was present, a precipitate appears immediately, or on boiling. The precipitate given by hydrosulphate of ammonia should be white if tinged with black, *iron* or *lead* is present.

Starch is said to be occasionally added, which, of course, will turn a deep blue with tincture of iodine.

If *baryta* and *lime* are present, and it is desired to ascertain whether there is *magnesia* also, see the

appropriate paragraphs in I., on the separation of baryta, lime, and magnesia.

To detect soluble forms of *alumina*, add to the muriatic acid solution ammonia in excess. The oxide of zinc at first thrown down, redissolves in the excess of ammonia, but alumina remains. If iron or lead be present, the colour of the sample will be yellowish.

§ 55. CARBONATE OF ZINC (*Calamine*).

Commercial calamine has often the following compositions :—

I.

Carbonate lime	3.6
Peroxide iron	5.2
Sulphate baryta	89.3
Water	1.9
	<hr/>
	100.0

II.

Carbonate lime	5.8
Peroxide iron	2.8
Sulphate baryta	85.2
Water	6.2
	<hr/>
	100.0

III.

Carbonate lime	5.6
Peroxide iron	3.2
Sulphate baryta	82.8
Water	8.4
	<hr/>
	100.0

IV.

Carbonate lime	1.3
Peroxide iron	1.4
Sulphate baryta	90.8
Water	6.5
	<hr/>
	100.0

V.

Carbonate lime	5.0
Phosphate iron	2.8
Peroxide iron	3.7
Sulphate baryta	84.8
Water	3.7
	<hr/>
	100.0

VI.

Silica	28.8
Oxide zinc	58.6
Carbonate lime	2.8
Phosphate iron	6.6
Water	3.2
	<hr/>
	100.0

The above were all offered for sale by "the most respectable drug-houses in London."

To examine samples of calamine, dry at a gentle heat, and note loss, *moisture*. Determine in a part of the dried matter, *carbonic acid* (I. § 8, *b*). Dissolve another portion of the dried substance in muriatic acid, and note the undissolved matter.

To the solution add excess of ammonia, *oxide of iron* is precipitated; zinc and lime remain in solution.

The *lime* may be thrown down from the ammoniacal

solution by oxalate of ammonia. The zinc remains in the filtrate, and may be precipitated by carbonate of soda; the precipitate washed, dried, ignited, and weighed as *oxide of zinc*; of which 40·2 parts denote 62·2 of pure carbonate of zinc. Carbonate of zinc should contain 64·63 per cent. oxide of zinc, and 35·37 carbonic acid.

§ 56. CHLORIDE OF ANTIMONY (*Muriate of Antimony*, or *Butter of Antimony*).

This substance is generally sold in solution containing more or less free hydrochloric acid. The amount of antimony present is determined as in II. § 158. The principal impurity to be noticed is chloride of iron. To ascertain whether this is present, add a large excess of water, which will precipitate the bulk of the antimony; then add hydrosulphate of ammonia, which will give a black precipitate if iron is present.

§ 57. TARTAR EMETIC (*Bitartrate of Antimony and Potash*).

The amount of antimony in tartar emetic may also be determined as in II. § 158. The principal impurity in commercial samples is bitartrate of potash (cream of tartar), which may be detected by the use of the following test liquid. Mix 8 parts acetate of lead, 15 parts acetic acid of specific gravity 1·064, and 32 parts of water. This liquid, added to a solution of the suspected sample, will cause a precipitate if cream of tartar be present. The precipitate requires a little time to form if the quantity of the adulteration be not very large.

§ 58. NITRATE OF SILVER.

Dissolve part of the sample in a little water, add hydrochloric acid, which will precipitate the silver as chloride. Filter, and treat the precipitate with excess of ammonia. If it all dissolve it consisted merely of chloride of silver; but if a part remain insoluble in the ammonia, chloride of *lead* is also present. Through the filtrate pass a current of SH. No precipitate should be formed if the sample be pure, but a brown or black precipitate may arise from *copper*, or from some chloride of lead, which remained dissolved in the muriatic acid and water. To decide as to the presence of copper, dissolve another portion of the sample in water, and remove the silver by adding hydrochloric acid as long as any precipitate is formed, concentrate the clear liquid, and add a few drops of yellow prussiate of potash. A chestnut brown precipitate will be formed if copper is present. Next take the liquid, through which SH has been passed, filtered from any precipitate of lead and copper, evaporate it to dryness, and ignite. Nothing should remain. A white saline residue shows the presence of nitrate of potash in the sample, which often occurs to a serious extent.

The stick or roll nitrate of silver is not to be relied upon, but that sold in crystals is generally pure.

§ 59. LAKES, VARIOUS (*Compounds of alumina or oxide of tin, with the colouring matter of cochineal, madder, lima and sapanwood, anatto, &c.*)

If an effervescence is caused by adding a dilute

acid, examine whether the carbonate present is *soluble* in boiling water. If it is, extract 100 grains of the sample with hot water, and determine *carbonate of soda* in the filtrate (I. § 8, *b*; II. § 1). If insoluble—the more probable case—determine carbonic acid in a weighed quantity of the sample, and note the result as *carbonate of lime and chalk*, 22 parts carbonic acid representing 50 parts carbonate of lime.

A fresh portion of the lake may be ignited to destroy organic matter; the residue is weighed and treated with a large excess of dilute muriatic acid. Precipitate the clear liquid with ammonia, filter off the precipitate of alumina, and in the filtrate determine lime (I. § 10). If there is more lime than will combine with the carbonic acid just determined, or if no insoluble carbonates have been found, the sample is adulterated with gypsum. The residue, insoluble in muriatic acid, may contain binoxide of tin, sulphate of baryta, and china clay. Wash and dry this residue, and heat it in a current of hydrogen gas, or heat it strongly before the blowpipe upon charcoal, mixed with some cyanide of potassium. In either case the oxide of tin is reduced, and may be afterwards dissolved out in a little muriatic acid. The china clay and sulphate of baryta may be separated as in I. § 91–94, but for all practical purposes they may be weighed together, and accounted for as *rank rubbish*.

§ 60. VERDITER, GREEN AND BLUE (*Mineral Green*
and *Mountain Blue*).

Ignite a weighed sample strongly; carbonic acid

and water are expelled, and black oxide of copper remains *alone*, if the sample be pure, to the extent of 69·5 to 71·7 per cent. in the *green*, and about 69 in the blue variety. Weigh the residue, dissolve in nitric acid, and determine the copper either as in I. § 74, or by the method directed, II. § 152. If it prove deficient, test the filtrate for lime (copper having been first removed by HS if the volumetric method have been used), and estimate its amount. Cheap samples may contain 70 per cent. of chalk.

Verditer must not be confounded with verdigris; the former is a *carbonate*, the latter an *acetate* of copper.

§ 61. BRUNSWICK GREEN (*Hydrous Oxychloride of Copper*).

Determine as above. If pure, it should contain 71·6 per cent. of copper.

§ 62. CHROME GREEN (*Chromate of Copper*).

Boil in caustic potash or soda. Filter, wash, and redissolve the precipitate, and determine *copper*. In the original alkaline filtrate determine *chrome* (I. § 52). Lime in various forms, and alumina, are the probable impurities. *Zinc* may also occur in colours made from impure sulphate of copper. Wherefore, in determining the copper volumetrically, the copper should first be precipitated with zinc or iron, and the precipitate redissolved as in II. § 152.

§ 63. SCHEEL'S OR SWEDISH GREEN (*Arsenite of Copper*).

Separate by means of soda-ley, and determine copper and arsenious acid (II. § 152 and 43).

Parrot green is the same article, with the addition of starch.

Neuwied green contains gypsum. Pickle green is a mixed carbonate and arsenite of copper. In all these the arsenic (present as arsenious acid) may be determined by means of nitrate of iron (I. § 88), or volumetrically.

If the sample is treated with excess of ammonia, the gypsum, alumina, and silica used as adulterants, remain undissolved, and may be weighed, whilst the arsenic and copper are dissolved.

§ 64. RED LEAD.

Digest in very dilute nitric acid mixed with sugar. Oxide of iron, brick-dust, and red ochre remain undissolved.

§ 65. LITHARGE.

Contains the same impurities, as also silica, which are determined in the same manner. Copper, rarely present in any extent, and of course never added purposely, is detected by digesting the sample in nitric acid, and adding ammonia in excess. If a blue tint appears in the solution it is filtered, and the copper determined in the filtrate volumetrically.

§ 66. CARMINE.

Extract a weighed portion with ammonia, wash the precipitate, and dry in the water-bath. The impurities—starch, alumina, chalk, vermilion, &c.—remain behind.—(See II. § 60.)

§ 67. VERDIGRIS, BLUE AND GREEN (*Bibasic and Sesquibasic Acetates of Copper*).

The former should contain 43·5, the latter 49·8 per cent. of copper. The sample should be completely soluble in ammonia. *Lime* in various forms will remain. Determine sulphuric acid in the liquid obtained by digesting 100 grains in water, and filtering. Its presence indicates *sulphate of copper*.

§ 68. SCHWEINFURT, OR VIENNA GREEN (*Aceto-arsenite of Copper*).

It should contain 31·5 per cent. of copper, 58·5 of arsenious acid, and 10 of acetic acid. Examine for chalk, gypsum, and alumina.—(See II. § 63).

§ 69. ULTRAMARINE.

Weigh off equal quantities of the samples. Make up a standard acid by diluting 1 ounce concentrated pure sulphuric acid with 20 ounces water, and find with the pourette the relative quantities of the acid required to convert the blue colour into a reddish grey.

§ 70. SMALT (*Powder Blue*).

Treat the samples with the standard acid mentioned in the last section. If genuine, they will be unaffected; if, as is frequently the case, they are made up of a carbonate (lime or soda), coloured with a little ultramarine of inferior quality, they will be decolorized with effervescence. For the determination of cobalt, see I. § 35.

§ 71. VERMILION.

Ignite a known weight. *Oxide of iron, red lead, and brick-dust* remain behind.

Take a fresh portion, digest it with nitric acid and a little muriatic until perfectly decomposed. Filter, supersaturate with ammonia, add hydrosulphate of ammonia in excess, and set aside for a day in a warm place in a loosely stoppered flask. Filter, dilute largely with water, acidulate slightly with acetic acid, and let stand in a warm place till all odour of HS has disappeared. Filter off the precipitate on a counterpoised filter, wash, dry at a gentle heat, and weigh. In the precipitate thus obtained, determine the amounts of sulphuret of arsenic and of sulphur, by digestion in excess of oil of turpentine, which dissolves the latter.

Vermilion in varnishes, mixed pigments, &c., may be detected by moistening the suspected substance with a solution of nitrate of silver containing an excess of ammonia. It instantly turns black.

§ 72. WHITE LEAD (*Ceruse*).

1. Digest a portion in excess of dilute nitric acid. Filter, wash, dry, and weigh the undissolved portion—*sulphates of baryta and lead*. To separate these treat the residue with tartaric acid, to which ammonia is added in slight excess. Sulphate of lead dissolves, sulphate of baryta remains.

2. Pass through the nitric acid solution a current of HS, and filter off the precipitate of sulphuret of lead.

In the filtrate *baryta* and *lime* (arising from carbonates of these earths present in the sample) are determined as in I. § 11.

The following recipes for white lead are in vogue amongst the "striking" class of colour-makers:—

COMMON WHITE LEAD.

Pure white lead, 1 cwt.; sulphate of baryta, 2 cwt.; chalk, 3 cwt. Mix.

DUTCH WHITE.

Sulphate of baryta, 28 lbs.; pure white lead, 84 lbs. This is a shade better.

GERMAN WHITE LEAD.

Pure white lead, 1 cwt.; sulphate of baryta, 2 cwt.; and a variety of such mixtures, differing merely in proportion.

§ 73. CHROME YELLOW, CHROME ORANGE, AND
CHROME RED.

Treat a portion in fine powder with hot muriatic acid, adding alcohol from time to time. Concentrate the green liquid, and filter. The insoluble residue is washed with dilute alcohol, and the washings added to the filtrate, in which chrome is determined as in I. § 52. Every 4 grains of chromic oxide represent 16.4 grains of *pure* chrome yellow. The ordinary impurities are sulphates of lead, baryta, and lime. The lightest samples are the worst.

§ 74. CHROMATE OF ZINC.

Treat a portion with excess of ammonia, the impurities remain undissolved.

§ 75. PRUSSIAN BLUE.

Carbonate of lime is betrayed by the effervescence arising on the addition of a dilute acid.

Sulphate of lime, and porcelain clay are left undissolved after the prussian blue has been extracted with one-sixth its weight of oxalic acid dissolved in water.

Starch is known by its swelling up and forming a jelly, when the sample is treated with boiling water.

Turnbull's blue is subject to the same impurities, and is examined in the same manner.

§ 76. DRY COLOURS (*General Method*).

The following very simple method will, in most cases, serve for determining the relative value of dry colours. Reduce a portion of the sample in question, to very fine powder, and lay it upon a sheet of paper. Take also a *perfectly pure* specimen of the same colour, (if you can get one) likewise in fine powder, and observe how much of any white powder, *e. g.*, finely ground gypsum or chalk, is needful to let down the pure sample to the same intensity of shade as the sample under examination.

§ 77. MADDER.

To determine the mineral impurities contained in madder—brick-dust, yellow ochre, sand, or clay—a portion is very carefully dried at 212° Fahr. as long as it loses moisture. When perfectly dry, 100 grains are placed in a platinum crucible and ignited, till all

carbonaceous matter is destroyed. The amount of ash should not exceed five per cent. If the madder has been carelessly prepared, the ash may reach seven to nine per cent.; but in adulterated samples it sometimes amounts to twenty per cent.

Organic impurities, *e. g.* ground dye-woods, bran, almond shells, and sawdust, cannot be separately determined. It is therefore necessary to determine the amount of colouring-matter contained in the sample as follows :—

1000 grains of madder are mixed with 1000 grains of pure, concentrated, sulphuric acid, and allowed to stand for some hours at an ordinary temperature. The mass is mixed with water, thrown upon a filter, washed as long as the washings have an acid reaction, and then dried at 212° Fahr. When dry it is carefully pulverized, and steeped in cold alcohol mixed with a little ether to remove fatty matters. This process should be three times repeated, requiring altogether about two hours. It is next boiled in about 5000 grains of alcohol, at 0.834. This process is also thrice repeated, to extract every trace of colouring matter. The alcoholic extracts are mixed, and concentrated by distillation in a glass retort. When the residue becomes of a syrupy consistence, it is poured into a balanced porcelain capsule, evaporated to dryness on the water-bath, and weighed. The per centage of true madder colour is thus readily determined.

The method employed by merchants in judging of the quality of samples of madder, *i. e.*, placing small

heaps on a sheet of paper, and exposing them for twelve hours to a damp atmosphere, is not only fallacious, but gives direct encouragement to fraud. Samples purposely adulterated, have been pronounced by experienced brokers superior to others perfectly genuine.

Garancine may be at once extracted with alcohol, without any previous preparation, and determined as above.

Munjeet is examined as madder.

§ 78. COCHINEAL.

To find the relative value of different kinds of cochineal, twenty grains of each sample are finely powdered, and dissolved at a gentle heat in two ounces of a weak solution of caustic potash; four ounces of water are afterwards added, and the liquid is allowed to cool.

A pourette, graduated into one hundred equal parts, is made up with five grains pure ferridcyanide of potassium, dissolved in a sufficient quantity of water. This test-liquor is gradually dropped into the solution of cochineal, till the purple colour is destroyed, and a reddish-brown produced. The change of colour may be best observed by applying drops of the liquids with a glass rod to a white slab, or white paper.

The value of cochineals may also be determined by the colour-test as with indigo. The samples are boiled each in a quart of water, and equal portions of the liquids, when cool, are introduced into the tubes of the colorimeter.

§ 79. INDIGO.

The inorganic impurities in indigo, such as lime, sand, clay, powdered slate, &c., may be determined by igniting a weighed portion, previously dried, in a platinum crucible. The ash, in a genuine sample, should not exceed six to seven per cent., except in Senegal indigoes, which may contain twelve per cent.

To determine the relative value of a sample of indigo, it is necessary to ascertain how much indigo-blue it contains. Rub fifteen grains of indigo to a fine powder, add 150 grains of fuming sulphuric acid, and leave the mixture covered for six to eight hours, occasionally stirring. The whole is then poured into a strong evaporating basin, containing two pounds of water, and with the addition of 750 grains of strong muriatic acid, is heated to boiling, replacing from time to time the water which evaporates.

Meanwhile, four grains of pure dry chlorate of potash are dissolved in 1500 grains of water, and placed in a graduated pourette of sufficient capacity. This liquid is gradually added to the solution of indigo, which should be well stirred, and as the process approaches completion, boiled between each addition. The liquid changes gradually from blue to green, brownish-green, and finally to brownish-red. The operation is complete when the last tinge of greenish-brown has disappeared. To observe these changes more readily, a mark may be made with a glass rod dipped in the liquid upon white unsized paper. By comparing the number of degrees of

chlorate consumed by different samples, their relative value will at once appear; or—

Rub ten grains of the sample previously reduced to a very fine powder, with two measured drachms of fuming sulphuric acid. The mixture is placed in a flask with a lightly-fitting cork, and digested for twelve to fourteen hours with occasional stirring, at a temperature of about 80° Fahr. Some fragments of glass or porcelain may be added to prevent the indigo from clotting together. When the indigo is perfectly dissolved, it is gradually poured with constant stirring into a pint of water contained in a basin, three-fourths fluid ounce of concentrated sulphuric acid added, as also the rinsings of the flask.

A pourette of 100 divisions is now made up with seven and a half grains of pure dried bichromate of potash, and this liquid is added by degrees to the indigo solution in the basin, until a drop of the mixture placed on unsized white paper appears of a pale brown, without any blue or green shade. The number of degrees of bichromate used indicates the comparative value of the sample.

The best method of applying the test-drops to the paper, is to bring the end of a glass rod in contact with the solution, and then gently press it upon the paper. After each addition, the mixture should be well stirred and kept at a gentle heat.

There is some difficulty in deciding on the exact point of saturation.

A simpler method consists in observing the intensity of the colour yielded by dissolving a known weigh

of the sample in sulphuric acid. For this purpose, take twenty grains of each sample to be tested, place it in a small flask, add 400 grains of pure sulphuric acid of specific gravity 1.845, and leave the whole for about an hour at a temperature of 100° Fahr. When it is thoroughly dissolved and cooled, it is poured into a two-quart bottle, the flask well rinsed with water, and the rinsings added, and more water poured in until the bottle is full. 10,000 grains of the liquid are now poured into a beaker, which is set aside for a few hours, in order that any solid matter held in suspension (*e. g.* undissolved mineral impurities) may be deposited.

The colorimeter, or apparatus for comparing the tints of the several samples, is arranged as follows: A number of cylindrical glass tubes closed at one end are required. These must be of the same kind of glass, and exactly equal in bore and in thickness; about half an inch in diameter, and fourteen inches in length. These are divided by a mark into two equal parts, the upper half being graduated from 0° to 100° . A small wooden box is provided, blackened within, and furnished with two apertures above, through which the tubes are introduced. Behind are square slits exactly opposite the tubes, and corresponding to their diameter, and in front is a sight-hole.

Each tube being filled with one of the solutions up to 0° , the box is held up to the light, when, by looking through the sight-hole it is easy to distinguish which is the more intensely blue. To the deeper of the two water is added, and the tube well shaken to

insure perfect mixture. This is continued until the two appear exactly equal. The number of degrees of water added show the relative superiority of the more intense solution.

§ 80. LAC DYE.

Proceed as for cochineal.

Lac lake is examined in a similar manner.

§ 81. SAFFLOWER (*Carthamine*).

The value of a sample of safflower depends upon the amount of red colouring-matter which it contains. To ascertain this relatively, take a known weight of the sample, and remove the yellow colouring-matter by repeated washings in pure cold water, until the latter no longer acquires a yellow tinge. The residue is now treated with water in which a little carbonate of potash has been dissolved, as long as any colouring-matter is extracted. A known weight of cotton yarn, bleached and perfectly clean, is introduced into the liquid, which is cautiously neutralized by the addition of tartaric, citric, or phosphoric acid. The cotton is gently agitated in the liquid until all colouring-matter is taken up, when it is withdrawn, well washed in cold water, and the depth and purity of its colour compared with that yielded by an equal weight of other samples.

The liquid extract of safflower (*carthamine*) should be of a fine scarlet, not crimson hue; and if rubbed upon the finger-nail should leave a beautiful golden-green stain. It is scarcely capable of adulteration;

but different samples may be compared by dyeing with them known weights of cotton or silk.

• The colouring-matter of safflower is insoluble in water.

§ 82. QUERCITRON BARK (*Flavine*).

The respective value of different samples of quercitron bark may be best ascertained by comparing the depth and purity of colour produced by equal weights when extracted either with water, or with an alkaline solution, for which purpose the colorimeter will be useful.

• Samples of flavine may be dissolved in a solution of carbonate of soda, and compared in the colorimeter. Or, determine (II. § 95) the respective proportions of tannin. The sample containing the least of this principle is most valuable.

§ 83. LOGWOOD (*Ground*).

• Take a weighed portion, and dry it at a gentle heat. The loss indicates water, which is sometimes fraudulently added to a serious extent. Examine also whether the sample has an alkaline reaction, as urine is sometimes sprinkled upon ground dye-woods to brighten their colour.

• In the same manner examine *sapan, peach, bar, cam*, and other dye-woods.

§ 84. ANNATTO.

Dry a portion at 212° Fahr. The loss of weight averaging 68 per cent. is moisture.

Ignite the dried residue and weigh. The residue of mineral matter should not exceed seven to ten per cent. on the dried, or one to two per cent. on the undried substance. Adulterated samples sometimes contain oxides of iron, brick-dust, sands, ochres, &c., to the extent of 87 per cent. on the dried article.

§ 85. LICHENS (*Tinctorial power of*).

To test the dyeing powers of lichens, they may be dried, powdered, and steeped for a few hours in a temperature of 130° Fahr. in a weak, though still moderately pungent solution of ammonia.

§ 86. ARCHIL.

Put fifty drops of archil, and three ounces of water into a flask, acidulate slightly with acetic acid, and add fifty drops of a newly-made solution of proto-chloride of tin (one part tin crystals to two of water). Heat the flask on the sand-bath. If the archil be pure, the liquid will be immediately decolorized on reaching the boiling point, exhibiting only a sallow yellowish tint, with a sediment of the same colour. If the sample be adulterated with log-wood or sapan, a faint bluish-grey, or red tinge will remain.

“Red” archils are often contaminated with red prussiate of potash.

§ 87.

Extracts of Dye-woods are sometimes “sprung” as it is technically styled; *i. e.* mixed with saline matter to increase their specific gravity. To detect this,

evaporate a known quantity of the extract to dryness, and heat the dry residue to redness, continuing the heat until all organic matter is removed. The saline matter (generally some neutral salt of soda or potash, as least likely to interfere with the uses of the extract) will remain and may be tested. Note, however, that if a nitrate be present there will be deflagration, whilst the substance is being ignited, and the residue will have an alkaline reaction. Sulphates, if present, will in part be reduced to sulphurets; which, if moistened with a dilute acid, emit fumes that blacken paper steeped in acetate of lead.

It is often difficult to determine by test-paper the presence of slight traces of acid or alkali in dye-wood extracts, dye-baths, and other deeply-coloured liquids; the tinctorial matter masking or hiding the acid or alkaline reaction. In such cases let the blue litmus paper used be made of unsized paper, and very faintly tinged, and *float* it on the surface of the liquor to be tested. The acid will penetrate more rapidly than the colouring-matter, and will redden the upper surface. For alkalies, proceed in the same manner with red litmus paper.

§ 88. DYED AND PRINTED GOODS, DETECTION OF DYES IN.

a. Mordants.—Burn a portion to ashes; extract the ash with concentrated muriatic acid. Examine the matter left unattacked; a deep green powder soluble in concentrated sulphuric acid indicates *chrome*; a yellowish-white powder, scarcely attacked

by any reagent except fusing potash or soda, is oxide of tin.

The muriatic solution is concentrated to expel free acid. A blue precipitate on the addition of yellow prussiate of potash indicates *iron*; a white flocky precipitate on the addition of caustic potash, and soluble in excess, is *alumina*.

Lead will rarely occur except in *yellows*. Its presence is ascertained by digesting a piece of the stuff in hydrosulphate of ammonia in a flask, when it will be blackened.

b. Blue colours:—

Indigo, fixed in the *blue vat*, and as *China blue*, is untouched by dilute acids and alkalies, destroyed by chlorine, and then takes no colour if dipped into logwood.

Pencil blues have the same behaviour with acids, alkalies, and chlorine, but turn red if subsequently dipped in logwood.

Soluble indigo is destroyed by chlorine and by alkalies; leaving in the latter case a white ground.

Prussian blues are untouched by bleaching powder but destroyed by alkalies, leaving a rusty ground.

Logwood blues are destroyed by weak acids and turn red.

Mixed blues.—Logwood, if present, is first removed with a weak acid, and the tests for distinguishing indigo from prussian blue are then applied to the washed stuff.

Ultramarine remains unchanged in the ash; or, moisten the pattern with ether, and then with muri-

atic acid. The gas given off blackens paper dipped in the subacetate of lead.

c. Red colours:—

Safflower is discharged by caustic alkalies.

Madder.—Hydrochloric acid gives a yellowish or orange cast, which subsequent treatment with milk of lime turns into a violet; which, on boiling with soap, passes into rose colour. In topical madder reds the whites are coloured by logwood. Garancine red and rose give a dull blue, not a violet, on treatment with milk of lime after hydrochloric acid.

Brazil wood turns orange with strong sulphuric acid; and with milk of lime (after hydrochloric acid or tin-salt) a fugitive violet, which disappears on boiling with soap.

Cochineal turns a bright cherry with strong sulphuric acid.

d. Yellow colours:—

Quercitron, discharged by chlorine and sulphurous acid, not perceptibly turned orange by potash or tin salt.

Buckthorn berries, discharged by chlorine, turned orange with tin salt; stone colour with sulphuric acid.

Fustic and fustet (orange and nankeen) discharged by nitric acid, turned red by sulphuric acid; catechu colour with caustic potash.

Sumach yellows; red with nitric acid; sulphuric acid, little alteration; sulphate of iron, grey.

Anatto, discharged by nitric acid; chlorine little affected; strong sulphuric acid, bluish-green.

Chrome yellow, destroyed by caustic alkalies; unaltered by weak hydrochloric acid.

e. Blacks:—

Logwood blacks are discharged by chlorine, leaving a yellow ground; hydrochloric acid, cherry red.

Catechu blacks hydrochloric acid, a dull orange. Both these are discharged by bleaching-powder, which *Chrome black* is not, but turned a chestnut-brown.

§ 89. GUMS.

Arabic, senegal, tragacanth, and cherry give a yellow precipitate with sulphate of iron. Dextrine (British gum) gives no precipitate.

Arabic and senegal give, with the subacetate of lead, a white curdy mass; and with alcoholic tincture of guiacum a fine blue colour.

Tragacanth and cherry give with subacetate of lead a transparent jelly.

Tragacanth does not change colour with the tincture of guiacum.

The gums are not coloured by iodine (distinction from the starches); they are precipitated by alcohol from their solutions in water. The gums seem an intermediate stage between starch and sugar, into which dextrine can be converted by diastase, or by boiling with dilute acids.

§ 90. STARCHES.

Weigh out 200 grains, and place in the desiccating apparatus over sulphuric acid. Note the amount of moisture lost. Expose the dried portion to the air

again, and by weighing from time to time, ascertain whether the moisture lost is slowly or speedily taken up again.

N.B. A starch that readily absorbs moisture from the air is comparatively worthless to the laundress.

Determine the amount of actual starch present by boiling with dilute sulphuric acid, thus effecting its conversion into sugar, which is then determined as in II. § 91. 100 parts of pure dry starch should yield 122 parts of grape-sugar.

Examine as follows whether any nitrogenous organic body be present:—Dissolve 1 part of mercury in 2 parts of nitric acid of specific gravity 1.25 at a gentle heat. Boil the solution afterwards for a few minutes. If this liquid communicates a reddish colour to the sample, nitrogenous matter is present.

Examine carefully with blue litmus paper whether the sample has an acid reaction.

To determine the relative tenacity of starches weigh out 24 grains of each sample, and mix with 400 grains of distilled water in a capsule of suitable size. The mixtures are then heated and boiled briskly for three minutes, with constant stirring, and poured into conical test glasses (Clarke's pattern), which the jelly nearly fills. The time at which each glass was filled is noted, and exactly two hours are allowed for the sample to cool in a current of air. Then take a flat metal disc, 7-10ths of an inch in diameter, lay it upon the surface of the jelly, and add weights until the skin of the jelly is broken, and the disc begins to sink in. The disc should not weigh above 50 grains. The

weights required to tear different kinds of starch jellies according to the experiments of Shier, vary from 87 to 2446 grains.

The more tenacious starches are preferred, both as articles of diet, and for stiffening woven fabrics, provided, in the latter case, they are not defective in clearness.

The clearness of the different starch jellies may be observed by putting equal quantities of the samples prepared as above in tall glasses.

The true starches are insoluble in water, both cold and hot, but gelatinize in the latter. They are coloured dark-blue by a watery solution of iodine, and a fine yellow by bromine. They are perfectly insoluble in alcohol and ether. The starches do not reduce salts of copper and silver. Heat and dilute acids, convert them into dextrine (British gum), and glucose or grape-sugar. When heated with stronger nitric acid they yield saccharic, tartaric, and oxalic acids. On exposing the different starches to the vapour of iodine, that of *wheat* becomes violet; of *potato*, a lilac grey; of *arrowroot* (genuine), a bright chocolate; of *tapioca*, yellowish or pale buff; of *sago*, yellowish.

The best method of distinguishing the starches of different vegetables, is to examine with the microscope the size and shape of the granules. The granules of wheat and barley starch are nearly globular, those of canna, potato, sago, rye, tapioca, and millet, oval and irregular. Those of oat, rice, maize, and yam, angular.

The so-called lichen starch (of Iceland moss), and

inuline (of the dahlia and dandelion root), are distinguished from true starches by being *soluble* in hot water. The former is coloured a dirty green, and the latter a yellow, by a watery solution of iodine.

§ 91. SUGAR (*in commercial samples and vegetable juices*).

Prepare as follows a standard solution of copper :— Take of pure crystallized sulphate of copper, pulverized and thoroughly freed from moisture by pressure between folds of blotting paper, 617·75 grains, and dissolve it in 2471·04 grains of water. In another vessel dissolve in a little water 2471·04 grains neutral tartrate of potash, and add about 10,000 grains of a solution of soda at 1·12° specific gravity. Pour the sulphate of copper solution gradually into this, and dilute the deep blue liquid to 17,787 fluid grains. Every 154 fluid grains of this liquid will contain 5·34 grains sulphate of copper, and answer to 0·77 of a grain of dry grape-sugar. Put the standard solution in a bottle, keep it in a cool dark place, and test it before use by boiling a portion with four times its bulk of water. The solution should not grow turbid.

The solution of sugar to be tested must be so dilute as not to contain more than $\frac{1}{2}$ per cent. Put 154 grain measures of the standard solution into a porcelain dish, add four times its bulk of water, boil gently, and drop in the sugar solution from a pourette. When the precipitate appears a bright red, extinguish the flame, allow the precipitate to subside, and slope the

dish so as to show the slightest greenish-blue tint. For certainty pour a little of the clear liquid into a test-tube, add a drop of the sugar solution, and heat. If any of the copper salt remain undecomposed, a yellow red cloud will appear in the liquid. If this is the case return the contents of the test-tube to the dish, heat, and continue adding the solution of sugar till the reaction is complete. The clear solution, finally, if filtered while hot, should give a precipitate neither with sugar nor with ferrocyanide of potassium. The number of degrees of the sugar-solution consumed will enable its per centage to be calculated.

Cane-sugar, before being tested, may be converted into grape-sugar by heating with a little dilute sulphuric acid. Deep coloured vegetable juices are first clarified by boiling with a little milk of lime, and filtering over animal charcoal, the washings being added to the filtrate.

In mixtures of grape and cane sugars the amount of the former is first determined as above. A second portion is then boiled with sulphuric acid and tested for total sugar. From this amount deduct the grape-sugar previously ascertained; 198 parts of this residue represent 171 parts of cane-sugar.

The sugars of commerce are adulterated with sand, gypsum, chalk, and starch, which remain undissolved on treating a portion with cold water, and may be filtered off; with common salt and sulphate of soda, which may be detected by evaporating the filtered solution to dryness, and igniting the residue in a platinum crucible.

The true sugars are all soluble in alcohol, as well as in water, and are resolved into alcohol and carbonic acid by fermentation.

Grape or fruit sugar (glucose) gives, even in the cold, an orange precipitate of suboxide of copper (see above). Heated with chromate of potash, to which an excess of alkali has been added, it gives a bright green colour. A slip of white woollen tissue, previously soaked in bichloride of tin, turns a deep brown if moistened with a solution of glucose, and heated.

Its solutions turn the plane of polarization from right to left.

Cane-sugar does not reduce copper in the cold. May be converted into grape-sugar by heating with dilute sulphuric acid.

Turns the plane of polarization from left to right.

Mushroom-sugar resembles grape-sugar, but does not reduce copper.

Sugary matters *not* fermentable.

Mannite does not reduce copper; crystalline, yields saccharic and oxalic acids with nitric acid.

Glycyrrhizine, not crystalline; precipitated by sulphuric acid from its solution in water.

Dulcose crystallizes rather like mannite, but with nitric acid it yields mucic acid, which dissolves in concentrated sulphuric acid with a blood-red colour.

Glycerine, not crystalline.—(See II. § 103).

Lactine (milk-sugar) forms hard white crystals; ferments with great difficulty; reduces copper; yields glucose if boiled with dilute sulphuric acid, but with nitric acid it forms mucic acid.

Inosite (flesh-sugar), crystalline; does not reduce copper; not fermentible.

N.B. The two latter are very sparingly soluble in alcohol.

§ 92. BLACK-LEAD (*Graphite, Plumbago*).

Black-lead is extensively used for making crucibles, either alone or mixed with other materials; in anti-friction pastes for machinery; for brightening and defending from rust metallic surfaces, especially such as are exposed to heat; and for lead pencils.

Of late years it has been very extensively adulterated with common salt, lamp-black, sulphuret of antimony, and black micacious minerals, *e.g.*, micacious iron ore. The superior qualities, intended for the pencil manufacturer, are mixed with sulphur and china clay, and cemented into a mass with gum-water.

These adulterations are thus detected:—The sample is treated with water, which will dissolve out salt and gum, if present. Their quantity may be determined by filtering the residue, drying, and weighing. The loss is, of course, matter soluble in water. Salt and gum are not likely to be found in the same sample.

If the sample, on digestion in muriatic acid, give off fumes of SH , which blacken moist carbonate of lead, some metallic sulphuret is present. The nature of the sulphuret present may be determined by applying suitable tests to the filtrate. If, on adding excess of water, a white milkiness appears, which is not produced in another portion to which tartaric acid has been previously added, antimony is present. Its

quantity may be determined as in I. § 86. To detect lead, concentrate the filtrate by heat, until the excess of acid is mostly dissipated, and add a little sulphuric acid; evaporate to dryness at lowest possible temperature, and add water. A white precipitate, soluble in excess of hot concentrated nitric acid, shows lead (I. § 73).

Lamp-black, or any other form of vegetable carbon, may be determined with tolerable accuracy by placing a weighed portion in a platinum crucible, and keeping it at a moderate red heat, with constant stirring as long as any loss of weight takes place.

To detect sulphur, take a portion previously freed from metallic sulphurets by digestion in muriatic acid, and proceed as in II. § 8.

It is scarcely possible to ascertain the exact quantity in which iron-ores and clay have been added as adulterants, since iron, alumina, and silica exist in black-lead as natural impurities, in quantities differing from 0.33 to 47 per cent. The total quantity of such substances present may be determined as in I. § 91 to 94.

Clay may be detected by boiling a portion, previously freed from metallic sulphurets, in concentrated sulphuric acid. The clear liquid, if clay was present, will contain sulphate of *alumina*; for the means of detecting which earth, see Appendix.

§ 93. EMERY.

Break off fragments from the pieces to be examined, and reduce it to a uniform coarse powder, so as to pass through a sieve containing 400 holes to the square

centimeter. The instrument called a "diamond mortar" will be found useful for this operation, giving only two or three blows with the hammer each time between sifting, and returning the fragments to the mortar which do not pass through. Mix well, and take about 10 grains of the powder. To try its quality, take a circular plate of glass, about four inches in diameter, and a small agate mortar. The glass is weighed and laid on a piece of glazed paper, the emery powder is thrown on it little by little, and rubbed against the glass with the bottom of the mortar. The emery is from time to time brushed off the glass with a feather, and when the whole has been made to pass once over the glass, it is collected from the paper, and made to pass through the same operation three or four times, until it is reduced to an impalpable powder. The glass is then weighed, and the loss noted. It has been found that while sapphire wears away glass equal to 80 per cent. of its own weight, the best emery wears about 50. Neither the specific gravity nor the chemical composition of a sample of emery, give any insight into its abrasive power or effective hardness.

§ 94. MIXED WOVEN TISSUES.

Suppose a woven fabric composed of silk, wool, and cotton.

1. Steep a weighed portion in an ammoniacal solution of the protoxide of nickel. The silk dissolves, and its quantity is determined by weighing the washed and dried residue.

2. Submit the residue to the action of steam at a

pressure of 1 to 2 atmospheres. The wool dissolves out. Wash, dry, and weigh the residue ; or—

Boil in caustic soda the result is similar.

Cotton and linen, unbleached, may be separated as follows:—Wash well in boiling water, and dry, then lay in a mixture of 2 pints dried nitrate of potash, and 3 pints of common sulphuric acid. Let it soak eight or ten minutes, wash and dry, and treat it with ether mixed with some alcohol. The cotton dissolves out as collodion. Wash the linen in boiling water, dry, and weigh.

Phormium tenax is thus distinguished from hemp and flax. Moisten with chlorine water, pour off after a few seconds, and add a few drops of ammonia. The phormium turns a violet-red, hemp a pale rose, while flax remains colourless.

§ 95. TANNER'S BARK, CATECHU, SUMACH, DIVI-DIVI,
MYROBALANS, VOLONIA NUTS, GALLS, MIMOSA
BARK, AND BODIES CONTAINING TANNIN.

Take a weighed quantity (from 50 to 100 grains) of the substance to be tested for tannin, and boil for a few minutes with as much distilled water as will cover them. This is repeated four to six times, with constant stirring. The decoctions are poured off each time without straining, collected in a beaker, and allowed to cool, access of air being prevented by covering the beaker.

Meantime, a test solution is prepared as follows:—Dissolve 1 drachm of clean Russian glue in 4 ounces of

distilled water, and add 15 grains of pure powdered alum to the solution, and shake it well round. It may be kept for some time in a stoppered bottle preserved from the light. 155 grains of this solution represent 5 grains of pure tannin (tannic acid). In testing a sample a sufficient quantity of the gelatinous fluid is poured into a Schuster's alkalimeter, which is then weighed with its contents. The test fluid is then carefully *dropped* into the beaker containing the solution of tannin until, on the falling of a drop upon the surface, the characteristic ring of tannate of gelatine is no longer produced. The precipitate is then allowed to settle, which takes place in a few minutes, and the clear supernatant is tested for an excess of tannin as follows:—Take a plate of black glass, or of common glass, and with a glass rod make upon it a couple of spots of the clear fluid. Add to one of these a drop of gelatine, and to the other a drop of solution of tannin. If the former gives a precipitate, the liquid to be tested requires a further addition of gelatine; but if the latter the mark has been overshot. By frequently repeating this process, towards the end of the analysis great accuracy may be reached. When the operation is complete, weigh the Schuster's alkalimeter; the loss of weight shows the quantity of gelatine consumed, from which the amount of tannin present in the sample is easily calculated.—*Müller*. Or—

Add to the tannic extract solution of gelatine, wash the precipitate dry at 212° Fahr., and weigh; 10 grains of the precipitate represent 4 of tannin.

§ 96. LARD, TALLOW, AND PALM OIL.

A known quantity of the lard is exposed to heat in the water-bath as long as vapour escapes. The loss of weight indicates water.

If a portion of the lard turns blue or black when rubbed in a mortar with tincture of iodine, starch or flour is present.

Let a quantity of the lard be carefully melted, and allowed to stand for a short time at such a temperature that it may not solidify. The clear liquid is then poured off and the sediment heated in a platinum crucible. It may consist of common salt, carbonate of soda, alum, or lime.

Tallow and palm oil being cheaper are less frequently adulterated, but may be examined in the same manner. Palm-oil often contains sand as an accidental impurity, the amount of which may be determined by melting and straining through lawn.

§ 97. ROSIN.

Inferior, dark-coloured samples are sometimes made to take up a considerable quantity of water, which adds to their weight, and improves their colour. Expose an ounce of the suspected sample to a steam or water-bath; heat and note the loss of weight and change of colour.

The presence of rosin in mixtures of organic, or of organic and inorganic matter, is generally detected by the scent, especially if the article be slightly warmed.

§ 98. OILS, FIXED.

The purity of fatty oils may generally be ascertained by Fischer's oleometer. This is a kind of hydrometer graduated from 22° upwards to 50°, the lowest degrees answering to the heaviest oils. In pure rape or olive oils this instrument stands at 37° to 38°, in poppy oil at 32° to 33°, in camelina at 31° to 32°, lintseed at 29° to 30°. As the specific gravity of oils is very much affected by temperature, a thermometer is inclosed in the stem, on which 0° answers to 60° Fahr. If the temperature be *higher*, the degree of the thermometer is subtracted from the degree shown on the hydrometer scale; if *lower*, it is added. The specific gravity of the chief commercial oils at 60° Fahr. is as follows:—

Tallow oil.....	0.900	Poppy.....	0.924
Rape.....	0.913	Camelina.....	0.925
Olive.....	0.917	Hempseed.....	0.926
Train.....	0.923	Almond.....	0.932
Cod	0.923	Lintseed.....	0.934
Walnut.....	0.923		

Castor and rosin oils are the heaviest, going beyond the range of Fischer's instrument. The use of the latter in adulterating more valuable oils, is easily detected, both by the increase in specific gravity, and by its peculiar smell.

The following tests will be useful. Rape, camelina, colza, or mustard oils, may be detected in other oils (*e.g.*, olive), as follows, even in quantities as small as 1 per cent. :—Boil in a porcelain dish from 400 to 500 grains of the sample along with a solution of 30 grains

REACTIONS OF

Oils.	Caustic Soda 1·34.	Sulphuric acid 1·475.	Sulphuric acid 1·53.	Sulphuric acid 1·635.	Nitric acid 1·18.
OLIVE.....	Faint yellow.	Green tinge	Greenish white.	Light green	Greenish.
GALLIPOLI.....	(Thick) do.	Ditto.	Grey.	Brown.	Ditto.
INDIA NUT.....	(Thick) white.	..	Dirty white	Light brown.	..
RAPE (pale).....	Dirty (thick) yellowish white.	..	Pink.	Brown.	..
POPPY.....	Ditto.	..	Dirty white
FRENCH NUT....	Ditto.	Brown.	Grey.	Brown.	Yellow.
SESAME.....	Ditto.	Green tinge	Greenish dirty white	..	Orange yellow.
HEMP.....	Thick) brownish yellow.	Intense green.	Intense green.	Intense green.	Dirty green
LINTSEED.....	(Fluid) yellow.	Green.	Dirty green	Green.	Yellow.
LARD.....	Pinkish white.	Dirty white	Dirty white	Light brown.	..
NEAT'S-FOOT....	Dirty yellow white.	Yellow tinge.	Brownish dirty white	Brown.	Light yellow.
SPERM.....	<i>Dark red.</i>	Light red.	Red.	Deep brown	Pale yellow
SEAL.....	Ditto.	Ditto.	Ditto.	Ditto.	Pink.

REMARKS.—*Caustic soda*, specific gravity 1·34, is used by adding 1 volume to 5 volumes of oil, mix and stand fifteen minutes. *Sulphuric acid*, at specific gravity 1·475, 1 volume to 5 of oil, mix and stand fifteen minutes. *Nitric acid*, at 1·18, same proportions, mix and stand five minutes. the proportion of 10 volumes to the 5 volumes oil, *after* the action of 1 volume nitric acid on the former at 1·845, the latter at 1·33; 1 volume of the mixture to 5 of oil; stand to stand five hours; 1 volume of this mixture is mingled with 5 volumes of oil, and allowed to stand five hours.

Particularly characteristic reactions are printed in italics.

lphuric d 1·635.	Nitric acid 1·18.	Nitric acid 1·22.	Nitric acid 1·33	Caustic soda 1·34.	Phosphoric acid, sir.
by gr an oil at 29 aff th te is sc th nse en. en. t wn. vn. th lat detto. by	Greenish. Ditto. Yellow. Orange yellow. Dirty green Yellow. .. Light yellow. Pale yellow Pink.	Greenish. Ditto. Yellowish red. Red. Ditto. Dirtygreen- ish brown. Yellow. .. Light yel- low. Ditto. Light red.	Greenish. Ditto. Red. Dark red. Ditto. Dirtygreen brown. Green, then brown. Very faint yellow. Light brown. Red. Ditto.	Fluid white mass. Fibrous white mass. Ditto. Fluid white mass. Light red fluid mass. Fibrous red mass. Fluid red mass, brown liquid beneath. Fibrous light brown mass. Fluid yellow mass. Fluid mass. Fibrous white mass. Fluid mass. Ditto.	Faint green Ditto. Brown, low. .. Green. Brown, low, green Dark red turns blue Ditto.

; 1 volume to 5 volumes of the oil, mixing and heating to boiling. It solidifies.
Sulphuric acid, at 1·53, same proportions, mix and stand five minutes.
Nitric acid, at 1·22 and 1·33, same proportions and time.
Phosphoric acid, 1 volume to 5 of oil. Mix 1 volume of 1 volume nitric acid at 1·33. *Aqua regia* 25 volumes muriatic acid
 1 p 5 of oil; stand two minutes. *Caustic soda* is used after aqua
 regis of oil, and allowed to stand five minutes.

Sulphuric and nitric.	Aqua-regia.	Caustic soda 1·34.
Orange yellow.	..	Fluid white mass.
Dark brown	..	Fibrous yellow white mass
Orange white.	..	Fibrous white mass.
Dark brown	..	Fibrous yellow white mass
Faint yellow.	..	<i>Fluid intense rose mass.</i>
Dark brown	Yellow.	Fibrous orange mass.
<i>Green, then intense red.</i>	Ditto.	Fluid orange mass with brown liquor beneath.
Green, then black.	Green.	Fibrous light brown mass.
Ditto.	Green, yellow.	Fluid orange mass.
Brown.	..	Fluid pink mass.
Dark brown	Faint yellow.	Fibrous brown yellow mass.
Ditto.	Ditto.	Fluid orange yellow mass.
Ditto.	Ditto.	Ditto.

the oils marked *thick*. *Sulphuric acid*, specific gravity 1·635, same proportions; mix and add *Caustic soda*, 1·34 specific gravity, is added in equal volumes of *Sulphuric* and *Nitric* acids equal volumes; 1·15, and 1 volume nitric acid at 1·33, allowed to stand precisely as after nitric acid.

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pure potash dissolved in 300 grains of distilled water. After boiling a few minutes pour upon a moistened filter, and test the liquid that flows through with a solution of nitrate of silver or subacetate of lead. If a black precipitate is formed, some one of the above-mentioned oils (all of which contain sulphur) is present. Or, the mixture of oil and potash may be boiled in a small silver cup, which will at once be blackened if a sulphuretted oil is present.

The fish oils are all blackened, if a current of chlorine be passed through them, a change not produced in the vegetable oils and in lard.

The drying oils, such as lintseed, nut, and poppy, may be distinguished from the non-drying rape, olive, &c., by the far higher temperature developed by the former in contact with sulphuric acid. Put into a small beaker about 2 grains of the sample to be tested, note the temperature with a slender glass thermometer, and add about 140 fluid grains of the strongest sulphuric acid. Stir the mixture with the thermometer, and note the rise of temperature. In a sample of pure olive oil the rise amounted only to 75° , whilst in poppy oil it was 135° .

The presence of any portion of drying oil is very objectionable in any oil used for lamps, in lubricating machinery, or in spinning wool. The non-drying are equally objectionable in varnishes, paints, &c.

Each kind of oil, when gently heated, emits a peculiar odour, very characteristic in case of lintseed, rape, fish, and rosin. As adulterators, however, add

substances to mask these odours; the test is not to be relied on singly.

Penot adds to 20 drops of the sample in question 1 drop of concentrated sulphuric acid, or of bichromate of potash dissolved in sulphuric acid, and notes appearances produced, as shown in the appended table :—

Oil.	Sulphuric Acid.		Bichromate of Potash.
	Not Stirred.	Stirred.	
OLEIC ACID..	Red spots and circles.	Reddish brown.	Reddish brown.
ALMOND	Yellow with orange spots.	Dirty green.	Yellow granules.
TRAIN.....	Red granules on brown ground.	Lees of wine.	Brown red granules on a brown ground.
HEMP.....	Brown granules on a yellow ground.	Greenish brown.	Yellow granules on green ground.
RAPE.....	Faint spots, green.	Green.	Yellow granules on green ground.
LINTSEED....	Reddish brown.	Brown lumps on green or grey.	Brown granules on a faint green ground.
NUT.....	Yellow.	Dirty brown.	Brown clots.
OLIVE	Yellow.	Brown grey.	Brown.
POPPY.....	Yellow spots.	Olive green.	Yellow granules on a pale green.

In testing oils, the same experiment should always be performed on a perfectly pure sample for comparison.

Rosin, or "pine," oil is now very extensively manufactured, and used for lubricating coarse machinery, and for adulterating more valuable oils. Its price allows of its being introduced even into lintseed oil.

Rosin oil has a turbid opalescent appearance; its colour is a reddish-brown by transmitted, and a lilac-brown, sometimes even a beautiful bluish plum, by

reflected light. It has a strong characteristic odour of rosin, by which it may be detected, except present in very small proportions. It is the heaviest of the fatty oils of commerce, having, at 60° Fahr., the specific gravity 0.985 to 0.990. The only oil approaching it in weight is castor oil, which, from its high price, is never used for adulterating other fatty oils. It lies beyond the range of Fischer's oleometer. It will, consequently, increase the specific gravity of any ordinary oil to which it is added. Thus, a lintseed oil containing 10 per cent. of rosin oil, will have the specific gravity 0.939 instead of 0.934.

If one drop of strongest sulphuric acid is added to 20 drops of the oil without stirring, there appears a yellow blot full of very fine orange stars and reticulations. The colour deepens into orange-brown and red-brown.

If stirred, the same proportions give a yellowness which changes into a turbid chestnut-brown, slightly mingled with grey at the edges.

With sulphuric acid at 1.475° specific gravity there is very little action, a faint fawn colour deposit, and round spots or stars of a rather darker shade.

Sulphuric acid at 1.635° specific gravity gives an immediate brown-red deposit—yellow in very thin layers—with abundance of very small round spots. In no case does sulphuric acid give any shade approaching to green.

Additional Remarks.

It is easy to ascertain from the state of the markets

what oils will be used for adulterating more expensive kinds. Olive oil is generally adulterated with rape; poppy and nut with lintseed. The two great classes of drying and non-drying oils are generally kept asunder by all dealers who value their reputation, since the addition, *e.g.*, of lintseed to rape, or of rape to poppy, will inevitably give dissatisfaction to the consumer, even though he may be unable to state the true cause of the evil. As long, however, as the demand for oils and fats appears to augment more rapidly than the supply, so long will dealers be strongly tempted to concoct fraudulent mixtures. The following is an American recipe for factitious lintseed oil:—

“Codfish oil, 100 gallons; ground litharge, 14 pounds; sulphate of zinc, 2 pounds. Boil together for two hours, cool a little, and add soap, 3 pounds dissolved in 1 gallon water. Mix well, and add boiled oil 8 gallons, and turpentine 2 gallons.”

In this country rosin oil—which is sometimes detected in samples of lintseed—will take the place of codfish oil.

§ 99. SPERMACETI.

A considerable part of the spermaceti sold is made up after the following or some analogous receipt:—

“Take white spermaceti, 10 parts; best stearine, 20 parts; potato starch, 5 parts; mucilage of gum, 1 part. Melt the two first, and unite them well with the starch while hot. Let the mass cool till it is of the consistence of dough; turn it out on a table covered with sheet lead, and roll it out thin; next sprinkle a little of the mucilage upon it, double it, roll and sprinkle again. Repeat this

process as often as may be judged necessary; lastly, allow it to cool. If it has been properly managed it will, when broken up, flake like spermaceti."

This article would be at once suspected in the candle trade, but if used for ointments, pomades, &c., the fraud would be less apparent. To examine suspected samples dissolve in ether. Starch, farina, gums, and mineral impurities are left behind. Carefully evaporate away the ether. The residue should have a bright pearly lustre, and should melt at 120° Fahr. Stearine melts at 140° Fahr.

§ 100. SOAPS.

1. Determination of moisture. Weigh out about one hundred grains in thin slices, and keep it at a temperature of 300° Fahr. for two or three hours. This operation should be performed in a balanced capsule. The loss on weighing = *moisture*.

A better process is to place the sample in a small porcelain boat, which is introduced into a wide glass or porcelain tube. The tube is connected at one end with an aspirator, at the other with a tube filled with chloride of calcium. The part of the tube where the boat lies is then heated to about 300° Fahr., whilst at the same time a current of dry air is sucked through by the aspirator.

2. For the fatty acids, dissolve 200 or 300 grains in water, and add muriatic acid. Boil the whole in a balanced porcelain capsule, and let it stand until the fats congeal. Then make a hole in the crust which they form, pour out the liquid, and add fresh water

until all saline matter is removed. The amount of fatty acids may then be weighed after careful drying over sulphuric acid. Their nature may be also ascertained by observing the melting point.

3. Determine in a fresh portion the alkalies by the ordinary alkalimetical process. The alkali in hard soaps is soda; in soft soaps, potash.

4. Evaporate the liquid and washings of No. 2 to dryness to expel excess of acid. Divide into two equal portions; in the one determine chlorides, and observe whether any excess appear above the quantity corresponding to the alkali found in No. 3. In the other portion, sulphates, if present, are determined.

5. Dissolve another portion of the soap in excess of water. Sand or clay, if present will remain, and is thrown on a filter, dried, &c., and weighed.

N.B. If rosin or starch be present, they will be found mixed with the crust of fatty acids, No. 2. Starch remains if the fats are dissolved in a mixture of alcohol and ether. The rosin is recognized by its peculiar smell, and by the stickiness which it imparts to the fatty acids.

Soluble silica is determined by evaporating the clear solution to dryness and igniting. Any part of the residue which is insoluble in water or dilute acids is silica, which has been present in the soluble state.

Another method is as follows:—

Weigh out about a pound, put it in a saturated solution of common salt, and boil. Take out and weigh the cold mass. The loss indicates *moisture*.

Next weigh out fifteen grains of the sample (in shavings if hard; if soft come as near fifteen grains as possible, but note the exact quantity), put it in a beaker and add a small quantity of ether, and a rather smaller volume of pure acetic acid. The soap dissolves and two layers are formed, the upper containing ether and *fat* or *rosin* with a little acetic acid; the lower layer water, acetate of *alkali*, free acetic acid, any *chlorides*, *sulphates*, &c., together with *starch*, *glue*, and other organic impurities, if present, either dissolved or in suspension. *Sand*, *clay*, *steatite*, and other mineral admixtures remain at the bottom of the glass. The entire liquid is then poured off from the undissolved substances in the beaker into a large pipette, widened in the middle, and turned upwards at the lower extremity, so as to form a kind of separator. In this the liquids are allowed to remain till they have perfectly separated into two layers. Manage so that none of the ethereal liquid enters into the lower turned up extremity. This may be effected by closing the lower aperture with one of the fingers of the left hand in which the pipette is held while the liquid is being poured in, and only allowing by an occasional slackening the pressure, the liquids to sink as low as the *bend*, until they are completely separated. The beaker is washed with ether and water, and the washings are poured into the pipette. When the liquids are properly separated, the aqueous solution of the saline matter is allowed to run out into another beaker. Successive quantities of water are then added to wash the ethereal solution remaining in the

pipette free from all saline matter; the washings being of course added to the saline solution. This being accomplished, the ethereal solution of the fat, together with a small quantity of water which adheres to it, are poured back into the empty first beaker, and the pipette is washed out with a mixture of strong alcohol and ether. The beaker should have been accurately balanced beforehand. The ethereal liquid is then evaporated at 212° Fahr., until nothing remains but the fat, or resin. When a trace of water remains beneath the fat, it is very difficult to remove it by evaporation, and the addition of alcohol to the ether is a very appropriate means of obviating this difficulty. Where the smell of alcohol, ether, and acetic acid has become very feeble, the residue is weighed, and the weighing repeated after a longer continued action of the heat.

The fatty acid from fifteen grains of soap, forms a layer of such thickness, that by slightly inclining the beaker, which should be narrow, the bulb of a small thermometer may be introduced, so as to determine the melting point; by which means some idea may be formed of the kind of fat.

If any insoluble residue remained in the beaker after the soap was taken up in ether and acetic acid, it is dried and weighed, and its nature may, if desired, be determined.

The aqueous saline liquid which was separated from the ethereal solution of the fats is now put in a silver or platinum capsule, and carefully evaporated to dryness on the water-bath. The residue is weighed,

and ought not to suffer any further diminution of weight, if again heated on the bath.

The presence of *glue* or *gelatine* may be detected during evaporation by the appearance—*starch*, by the solution of iodine; *caseine* (rare in England), by the peculiar empyreumatic odour developed on the application of a sufficient heat. The total quantity of such organic impurities may be estimated by the loss of weight upon ignition. If the perfect clearness of the aqueous solution, the small residue left on evaporation, and its radiated crystalline appearance indicate that saline bodies alone are present, the residue may be ignited at once, and a previous weighing dispensed with. In this case the ash should contain very little carbon. After this has been perfectly burnt off, it may be examined to determine the quantity and kind of mineral matter it contains. Soluble silica will have become insoluble, and may be filtered off and weighed. The filtrate is examined for chlorides and sulphates, and their quantity determined, if it appear greater than may be accounted for merely by the impurity of the alkali used in the manufacture of the soap, or the introduction of common salt during the process. If these admixtures need no determination, the total amount of alkali present may be found by treating the ignited residue with a sufficiency of hydrochloric acid, evaporating to dryness, weighing, and calculating from the chloride of potassium or sodium thus found, the amount of potash or soda. If both potash and soda are present, see I. §§ 1, 2, 3; II. §§ 1, 11.

Liquid Soaps.—Evaporate a weighed portion to

dryness, and note weight of the residue, then proceed as above.

The average composition of some of the principal soaps of commerce is as follows:—

	Fat.		Soda.		Water.
White Marseilles	50·2	4·6	45·2 = 100.		
Marbled Do.....	64·0	6·0	30·0	"	
Castile.....	76·5	9·0	14·5	"	
"	75·2	10·2	14·3	"	
Glasgow white....	50·0	6·4	33·6	"	

Soft Potash Soaps.

	Fat.		Soda.		Water.
London soft.....	44	9·5	46·5 = 100.		
" "	42	9·1	39·2	"	
" "	39	8·8	52	"	
" "	46	8·5	46·5	"	
Belgian soft.....	36	7·0	57	"	
Scotch soft.....	47	8·0	45	"	
" "	34	9·0	57	"	
Rape oil.....	51·66 ...	10·0	38·34	"	
Olive oil.....	48	10·0	42	"	

Soap best suited for cleaning Madder Reds and Pinks.

Fat.....	59·23
Soda.....	6·77
Water.....	34·00

100·00—Calvert.

Soap for Madder Purples.

Fat.....	60·40
Soda.....	5·60
Water.....	34·00

100·00—Calvert.

Silk Dyers' Soap.

Fat.....	61.9
Soda.....	8.1
Water.....	30.0
	<hr/>
	100.0

Wool Scourers' Soap.

Fat.....	61.4
Soda.....	8.6
Water.....	30.0
	<hr/>
	100.0

The presence of rosin, glue, or gelatine in any of its forms is highly objectionable in soaps designed for manufacturing purposes. For the detection of glue see detection of nitrogen, II. § 136.

Both glue and rosin may generally be detected by the smell, if the sample be heated.

A well-made silicated soap is admirably adapted for cleansing wool and woollen goods; the presence of *soluble* silica in soaps intended for this purpose should not therefore rank as an adulteration.

§ 101. ESSENTIAL OR VOLATILE OILS. .

The essential or volatile oils are often adulterated with fatty oils, solid fats, resins, and balsams, besides alcohol, methylated spirits, and essential oils of a cheaper quality.

The fatty oils, solid fats, resins and balsams are detected by dipping a strip of paper into the sample, and allowing the essential oil to evaporate. If any of

these impurities are present, a grease spot remains, whilst pure volatile oils leave nothing behind.

If alcohol be present, the sample grows turbid on the addition of water. Fragments of fused chloride of calcium are wholly or partially dissolved if alcohol be present, whilst in pure oils they will remain unattacked.

Cheaper essential oils used to adulterate the more costly, may often be detected by moistening a strip of blotting paper with the sample, and allowing it to evaporate at ordinary temperatures. The smell of the inferior oil may often be recognized towards the end of the evaporation. Oil of turpentine may very easily be detected in this manner.

Let a slip of paper be moistened with the acetate of lead, and held over the fumes of hydrosulphate of ammonia till it blackens. Put a drop of the oil to be tested upon the paper, and heat it very gently. The black colour is discharged by oils of turpentine, mint, and lavender, but is unaffected by those of cassia, anise, and lemon.

The following are the characteristic properties of some of the more important essential oils:—

Oil of Anise solidifies below 50° Fahr., forms a solid compound with sulphuric acid. If iodide of potassium saturated with iodine is mixed under constant stirring with oil of anise, a thick gelatinous paste is formed, which, on the addition of six to eight times the volume of alcohol, when further washed with alcohol, becomes a dazzling white. Its specific gravity is 0.985. With iodine it quickly congeals

into a hard mass with rise of temperature and orange vapours. If heated with sulphuric acid it turns a fine purple red and hardens quickly.

Oil of Star Aniseed.—With iodine it gives a solid compound with little heat or vapour. With sulphuric acid, a solid mass of a dark blood-red when heated. With nitric acid, a thick balsam turning yellow. Sparingly soluble in six parts of alcohol.

Oil of Bergamot forms an opaque solution with alcohol; a clear solution in potash (oils of lemon and orange dissolve opaque in potash). With iodine it fulminates. Specific gravity, 0.87 to 0.88.

Oil of Bitter Almonds dissolves of a clear red brown without decomposition in sulphuric acid; gives crystals with an alcoholic solution of potassium. It thickens with ammonia and muriatic acid. Acid reaction.

Oil of Cajeput—pale green colour; forms with iodine a loose coagulum, which changes into a greenish-brown, dry, brittle mass. Nitric acid gives a reddish-brown colour with violent reaction. Specific gravity, 0.91 to 0.92.

Oil of Cinnamon.—The true Ceylon oil is more mobile, lighter, and congeals less easily than the spurious. Both kinds dissolve in an alcoholic solution of potash, with a clear orange-brown colour, then turn turbid, precipitate a heavy insoluble oil, and grow clear again.

Chromate of potash gives with the Ceylon oil, brown flakes in a solution which is not yellow; but with the false oil, a faint yellow colour and no flakes.

Hydrochloric acid gives a violet colour. Sulphuric acid gives a purple red; and if added in excess, forms with the Ceylon oil a solid hard mass, turning from green to black; with the false, a softer substance of a deep olive green. Nitric acid gives a white crystalline mass and a red oil. Specific gravity, 1.03 to 1.09.

Oil of Cloves forms a crystalline mass with an alcoholic solution of potash, and loses its odour; with ammonia, a buttery clot; with a little sulphuric acid, a dark blue colour; with excess, a blood-red mass. Chromate of potash decomposes it, yielding brown flakes. Nitric acid occasions rapid decomposition. The least volatile essential oil. Specific gravity, 1.055.

Oil of Copaiva fulminates more feebly with iodine than turpentine, requires more alcohol for solution, and yields with sulphuric acid a darker and browner orange. Nitric acid causes a kind of explosion, producing a crystalline compound, at first yellow, then blue, and finally green. Specific gravity, 0.91.

Oil of Chamomile (*Matricaria chamomilla*), dark blue, viscid; dissolves iodine *without* heat, unless adulterated with oil of lemon or turpentine. Nitric acid decomposes it, and on the addition of water, precipitates a substance smelling like musk. Specific gravity, 0.924.

Oil of Cubebs—viscid; a weak action upon iodine giving a violet solution; with an excess of absolute alcohol, forms a clear solution; in equal weights, turbid and flakey. Sulphuric acid gives a crimson; nitric acid with heat, a pale red, and deposits a resin;

with hydrochloric acid a crystalline compound. Specific gravity, 0·929.

Oil of Lavender.—Explodes with iodine, leaving a pungent and acid residue; alcoholic potash yields a clear solution with a dark orange-brown colour. With sulphuric acid it thickens and turns a deep red brown. Specific gravity, 0·72.

Oil of Lemon behaves with reagents very like turpentine. Specific gravity, 0·848 to 0·85.

Oil of Orange differs from the former only in smell.

Oil of Peppermint does not explode with iodine, but forms with it a uniform mass. Gives with chromate of potash a deep brown red coagulum, which, when shaken, turns flaky, and the solution loses its yellow colour. Nitric acid (quarter of the bulk) gives a purple red. Specific gravity, 0·902 to 0·91.

Oil of Roses (Attar or Otto).—The physical characters are little to be depended on; thus, the yellow colour, with a cast of green, and the point of congelation, may be readily imitated. Its odour also is so powerful that a certain quantity of any other oil of a somewhat similar odour may be added without sensibly diminishing that of the attar. The manner in which it crystallizes is of a certain importance. When, after having been liquefied by a gentle heat, it is allowed to congeal slowly, and at rest it remains as it were transparent, in consequence of the thinness and perfect transparence of its long and pointed crystals, which resemble the blades of daggers, and on varying their position as regards the light, these laminæ reflect all the prismatic colours.

When the attar has been adulterated with any non-crystallizable oil which requires that the property of congealing should be imparted by the addition of spermaceti, it presents, when solidified, a number of fine needles, which, however, are not transparent, but render the mass uniformly semi-opaque. But for this test to be of any value, the attar must be allowed to crystallize slowly, and at rest, otherwise it becomes nebulous and opaque, like that which has been adulterated.

The oil most frequently used for its adulteration is that of several species of pelargonium. The oils derived from the different species cannot be all identical, since Recluz describes one that is solid, whilst that met with in commerce is always liquid. The oil which I employed for comparison is called oil of geranium, and comes from Nice. I also examined some oil of rosewood which was distilled at Paris some years ago, and some of that found in commerce. Three reagents will serve to distinguish the pure attar; iodine, nitrous acid vapour, and sulphuric acid.

Test with Iodine.—A small quantity of iodine is placed in a small glass vessel with a wide mouth; around it are arranged watch-glasses containing one or two drops each of the samples of oil under examination, and the whole is covered up with a bell-glass. At the end of a few hours the vapour of the iodine is found to have condensed upon every part of the interior of the bell-glass, and upon the glasses containing the oils; but the margin of the watch-glasses containing the adulterated oils is much more coloured than that

of the glass containing the attar, and when the former becomes brown, the latter retains its natural colour. After some hours the vessel containing the iodine may be removed, and then, the effect still continuing, the attar remains white, whilst the other oils become perfectly black. On exposing the watch-glasses to the air, the iodine which has condensed upon the margin of those containing the attar is volatilized, and the glass becomes almost colourless, whilst the others retain their black colour. This test will serve, not only to determine the nature of the attar, but also whether any oil of geranium or rosewood is present or not.

Nitrous Acid test.—For this use a small glass vessel is set upon a plate; 150 to 200 grains of concentrated nitric acid are placed in it, and some copper turnings added. Around this are placed watch-glasses, containing each one, or at most two drops of the oils, and the whole is covered with a flat bell-glass. In a few minutes the oil of rosewood acquires a dark yellow colour, the attar nearly the same, whilst the oil of geranium takes an apple green colour, and retains it for some time.

This test will serve to distinguish the two oils when in a state of purity, and may even serve to recognize oil of geranium containing the attar, because of the yellow colour which it imparts to the latter; but it is evident that it cannot serve to determine the presence of oil of geranium in the attar of roses.

Sulphuric Acid test.—One or two drops of the oil to be tested is put into a watch-glass, the same number

of drops of the strongest sulphuric acid are added, and the two fluids mixed with a glass rod. All the oils are rendered more or less brown by this proceeding; but—

The attar of roses retains the purity of its odour.

The oil of geranium acquires a strong and disagreeable odour, perfectly characteristic.

The odour of oil of rosewood is increased, and becomes somewhat unctuous. It is not, however, characteristic. The commercial oil of rosewood acquires an odour distinctly like that of cubebs.

Of these three methods, that with sulphuric acid is by far the best for distinguishing the attar of roses from oil of geranium, and for distinguishing the second in the former. The iodine test is also very certain, but it requires considerably more time. The nitrous acid test will serve to recognize the two oils when not mixed, or even to detect the attar of roses in the oil of geranium, but not the latter when mixed with the former.—*Guibourt*.

The specific gravity of attar of roses is 0.832. Its solid portion is insoluble in alcohol, but dissolves in ether.

Oil of Rue.—Iodine slowly dissolves without outward marks of reaction, yielding a slightly viscid liquid. This distinguishes oil of rue from oils derived from the coniferae, the aurantiaceae, and most of the labiatae. Nitric acid slowly changes it into a thin greenish-yellow liquid. Chromate of potash gives no reaction. The alcoholic solution is turbid. Potash gives a red-brown, and sulphuric acid a darker brown. Specific gravity, 0.837.

Oil of Sassafras.—Sparingly soluble in alcohol. With iodine it forms a clear solution without thickening. With half its volume of sulphuric acid, a green colour (perfectly characteristic), which turns crimson when heated. A larger amount of acid yields a splendid amaranth-red. Nitric acid decomposes it without heat, forming a reddish-brown resin, which grows hard and brittle when heated. Specific gravity, 1·08 to 1·09.

Oil of Thyme.—Does not react violently with iodine.

Oil of Turpentine.—Violent reaction with iodine; often ignition. Ignites with chlorine gas, and with a mixture of sulphuric and nitric acids; neutral to litmus paper. Specific gravity, 0·86 to 0·87.

The essences or volatile oils may be divided into two groups; those of the one render ether turbid, those of the other having a similar effect upon benzine. Those oils which render ether turbid have no such effect upon benzine, and *vice versa*. If we mix an essence which renders the ethers turbid, with one which perturbs benzine, they mutually trouble each other, but the opacity soon disappears, and the mixture then disturbs both ethers and benzine.

GROUP I.

Perturbing ether.

Aniseed.
Wormwood.
Dill.
Angelica.
Seville orange.
Badian.

GROUP II.

Perturbing benzine.

Bitter almonds.
Spikenard.
Bergamot.
Basil.
Cinnamon, Ceylonese.
" Chinese.

GROUP I.—*continued.**Perturbing ether.*

Cedar.
 Sassafras.
 Lemon peel.
 Pure citron.
 Carraway.
 Cummin.
 Skirret.
 Capaiba.
 Celery.
 Camomile.
 Cardamum.
 Tarragon.
 Bitter fennel.
 Sweet fennel.
 Orange flower.
 Ginger.
 Juniper.
 Hyssop.
 Mace.
 Myrtle.
 Musk.
 Portugal orange.
 Parsley.
 Pepper.
 Rue.
 Savary.
 Savin.
 Turpentine.
 Valeriac.

Also,
 Benzine.
 Sulphuret of carbon.
 Chloroform.

GROUP II.—*continued.**Perturbing benzine.*

Calamus.
 Coriander.
 Cassia.
 Cubebs.
 Cajeput.
 Cloves.
 Rose geranium.
 Lavender.
 Flower of lavender.
 Cherry laurel.
 Laurel.
 Pure mint.
 Marjoram.
 Balm.
 Allspice.
 Patchouli.
 Pennyroyal.
 Attar of roses.
 Rosemary.
 Wild thyme.
 Sage.
 Semen contra.
 Thyme.
 Tamarind.
 Vetiver.
 Indian vervain.
 Wine.
 Wintergreen.

Also,
 Ethers.
 Alcohol.
 Wood spirit.

Nitro-benzine, and the artificial essences produce no effect. Those of the second group by distillation, lose

the property of rendering benzine turbid, but if an oil of this group contains any of the others, it will render ethers turbid, even if rectified.

Of the first group, turpentine produces the maximum effect.

In examining essential oils, it is highly important to have genuine samples at hand for comparison, especially, as in many cases, the quality can only be judged by a careful observation of the odour. It is not generally known that the sense of smell may be rendered temporarily more acute by filling the mouth with the coldest water procurable.

§ 102. WAX.

Bees-wax is adulterated with the sulphates and carbonates of lime and baryta, oxides and carbonates of lead and zinc, rosin, suet, tallow, flour, and starch.

Samples are occasionally met containing no wax, but made up according to the subjoined, or some similar formula:—Take yellow resin, 70 lbs.; suet, 35 lbs.; potato flour, 9 lbs.; turmeric flour, 5 lbs.; mix, and form into cakes as it begins to cool, rub over each cake when cold with some flour. A little lamp-black may be added if the colour is too bright.

The mineral impurities are detected on boiling in water, when they settle to the bottom; starches and flour are known by the water in which the sample has been boiled turning blue on the addition of iodine.

Rosin, suet, and tallow are most readily recognized by the odour given off during combustion. A piece of lampwick is coated with the sample kindled, allowed

to burn for some time, and then blown out. The peculiar scent of the rosin, or of the fats present, is easily detected.

Rosin and fats may also be detected by the use of chloroform. If more than 25 per cent. of a sample of wax be taken up by treatment with six to eight parts of this solvent, the sample is certainly adulterated.

§ 103. GLYCERINE.

Pure glycerine is a syrupy inodorous liquid, of a very sweet taste, of specific gravity 1.28 to 1.27. It may be mixed in any proportion with water and with alcohol. It is very sparingly volatile, and when heated yields pungent vapours of acroleine, which attack the eyes. It reduces solutions of copper. When mixed with 6 per cent. of water it has the specific gravity 1.252. If submitted to a red heat in a capsule nothing should remain. Should a residue appear, it may be taken up in nitric acid and tested for lead and copper, or if they are absent, lime, alkalies, and for other mineral impurities. Glycerine should not redden blue litmus paper; it should have no appreciable odour if a drop is rubbed on the hand. One part by measure should dissolve completely in one part by measure of alcohol acidulated with 1 per cent. of sulphuric acid, without giving any appreciable deposit, even after twelve hours (the deposit would be caused by lime). If diluted and boiled with solution of soda or potash, it should not be altered in colour, showing the presence of glucose.

The following substances are soluble in glycerine:—

Sulphur dissolves only to about $\frac{1}{2000}$. The solution blackens a leaf of silver.

Sulphuret of Potassium dissolves in about 10 parts of glycerine, forming an amber-coloured liquid.

Sulphuret of Calcium dissolves to about the same extent.

Iodine dissolves in 100 parts of glycerine, forming a beautiful reddish liquid.

Iodide of Sulphur dissolves in 60 parts of glycerine.

Iodide of Potassium dissolves in 3 parts of glycerine.

Biniiodide of Mercury dissolves in 340 parts of glycerine; and the double iodide of potassium and mercury in 15 parts. The protiodide is completely soluble.

Chloride of Mercury is soluble in 14 parts; the subchloride insoluble. Pure *quinine* dissolves in 200 parts, and sulphate of quinine in 48 parts cold, 40 of hot glycerine. *Tannin* dissolves in 6 parts of glycerine in the cold, and 4 parts with heat. *Tannate of quinine* is taken up by 130 parts. *Morphia* dissolves in 19 parts of cold glycerine, and in 10 parts with the acid of heat. The hydrochlorate and acetate of morphia are likewise more soluble in glycerine than in any other solvent. *Strychnia* dissolves in 300 parts of glycerine, and the nitrate in 20 parts. *Veratrina* dissolves in 96 parts, *brucia* in 70 parts, and *atropia* in 50 parts of glycerine. *Tartar-emetic* dissolves in 30 parts of glycerine. *Sugar* with heat, in 10 parts. *Gum-arabic* dissolves in one third part. The resins, properly so called, are insoluble in glycerine, but the so-called gum-resins are more or

less soluble. Camphor requires 400 parts for solution. *Starch* swells lightly but does not dissolve.

§ 104. GLUE ; LIQUID GLUES.

Glue is not subject to much sophistication. Mineral impurities, such as alum, which is sometimes added in a larger quantity than requisite, may be determined by incineration in a platinum crucible. The crucible should be placed in a slanting position, and be half covered with its lid to promote a current of air into the interior. Without this arrangement the combustion of any organic substance containing nitrogen is apt to be very tedious.

Glue should be perfectly soluble in hot water, without turbid floating matter or sediment. The relative tenacity of different samples may be ascertained by a process similar to that directed for starches.

Solutions of glue give with tannin a greyish-white precipitate ; chloride of mercury no precipitate ; sulphate of platinum brown viscid flakes. The acetates of lead give no immediate precipitate. The persulphate of iron gives no precipitate except on boiling. It is not precipitated by acids. Glue is insoluble in alcohol, ether, and chloroform. If heated with potassa and sulphate of copper a violet colour appears. The same reaction is produced by fibrine, albumen, and caseine, which, however, are not readily confounded with gelatine.

In examining *liquid glues*, of which several varieties occur in commerce, test for nitric acid, alum, acetic

acid, and alcohol. The volatile acids and alcohol are best separated by distillation in a glass retort at a gentle heat. Nitric acid rarely occurs in the same formula as acetic acid and alcohol. Sugar may also be sought for.

§ 105. ALCOHOL; METHYLATED SPIRIT; FINISH.

Pure alcohol has the specific gravity 0.791; if it gives a blue colour to the white anhydrous sulphate of copper (formed by carefully heating common blue vitriol), water is present. Essential oils, if present, may be detected by adding a little of a solution of nitrate of silver, and exposing the whole to direct sunlight, when it will turn red. It should be neutral to test-paper, and evaporate without leaving any fixed residue.

The same tests, except that for water, are applicable to the dilute or aqueous alcohols used for manufacturing and pharmaceutical purposes under the name of spirit of wine, containing from 50 to 70 per cent. of actual alcohol.

A mixture of alcohol and wood spirit is now sold under the name of "methylated spirit." The presence of methylic or wood spirit is ascertained by adding to the sample caustic potash in powder. If even one per cent. of wood spirit be present, the mixture will turn brown in half an hour.

"Finish" is a spirit holding a small amount of shellac or other resinous matter in solution. It is sometimes sold as spirit of wine, but the resin may be detected on evaporation.

"Fusel oil" is often present in whisky, and sometimes in spirit of wine. To detect it, put some chloride of calcium in small pieces into a beaker, pour in sufficient of the sample to moisten them, cover the beaker with a plate, and allow it to stand for a couple of hours. On withdrawing the plate and applying the nose, the rank disgusting smell of fusel is very perceptible.

Mixtures of alcohol and water at different strengths are denoted by commonly-received names; thus the "proof spirit" of the British excise has the specific gravity 0.92; and contains at 68° Fahr. about 47 per cent. by weight of actual alcohol. "Rectified spirit" has the specific gravity 0.90, and *spiritus vini rectificatissimus* about 0.83 to 0.84.

The subjoined table shows the amount of absolute alcohol in hydrated alcohol of different specific gravities at 68° Fahr.

Alcohol by Weight.	Water.	Specific Gravity.
100	0 ..	0.791
95	5	0.805
90	10	0.818
85	15	0.831
80	20	0.843
75	25	0.856
70	30	0.870
65	35	0.883
60	40	0.895
55	45	0.906
50	50	0.917
45	55	0.928
40	60	0.939
35	65	0.948
30	70	0.958

Alcohol by Weight.	Water.	Specific Gravity.
25	75	0·965
20	80	0·971
15	85	0·977
10	90	0·983
5	95	0·991
0	100	1·000— <i>Meissner</i> .

§ 106. ETHER (*Sulphuric Ether*).

Pure ether has at 68° Fahr. the specific gravity 0·713. It boils at 98°, and does not freeze at any temperature yet attained.

The ether of commerce is contaminated with water and alcohol, and has the specific gravity 0·78. The presence of water and alcohol may be detected by shaking it up with some oil of copaiba. If it be pure it remains clear; but if water and alcohol are present, it forms an opaque emulsion.

It should be perfectly neutral to litmus paper.

§ 107. CHLOROFORM.

The impurities present in chloroform are alcohol, aldehyde, ether, muriatic and hypochlorous acids, and certain methylated compounds. The genuine article should have the specific gravity 1·500; it should be perfectly transparent and colourless; should neither redden nor bleach litmus paper; should not become opaque when dropped into water; should not cause any white precipitate or turbidity in a solution of nitrate of silver; neither whiten nor coagulate fresh white of egg, even on prolonged contact; should not give a brown colour to a solution of caustic potash

when heated therewith, and should dissolve completely in alcohol and ether. When shaken with pure concentrated oil of vitriol of the highest strength, it should not become yellow or brown, but remain colourless and give no colour to the acid.

§ 108. ORGANIC BASES (*General Method*).

For the detection of organic bases or alkaloids in unknown mixtures, proceed as follows:—Mix with the substances a quantity of pure, dry, quick lime; if the reaction of the substance be not very powerfully acid, 12 per cent. of their weight will suffice. Grind all up in a mortar, heat at 212° Fahr. till perfectly dry, and grind very fine. Exhaust the powder three successive times with boiling alcohol, and filter after cooling. The filtrate will contain any organic bases present, along with, possibly, oils, fats, and resins.

Evaporate the alcohol slowly away, and put the cold dry residue into ether, which takes up oils and fats. If the alkaloid sought for be insoluble in ether (morphia, strychnia, brucia), it will remain suspended and may be separated by filtering or decantation, and submitted to special tests. If the alkaloids are taken up, the ethereal extract is cautiously evaporated to dryness, and the residue treated with water acidulated with sulphuric acid. From the solution the alkaloid is precipitated with ammonia and specially examined.

This method is open to objection in case of the volatile bases, nicotine, conicine, &c., for although their boiling points when alone are above 212° Fahr.,

yet when heated with moisture and a powerful mineral base, a portion may be dissipated; or—

31 The substance, if a liquid or paste, is mixed with twice its weight of pure and strong alcohol; if a solid, it is chopped or pounded into small fragments, moistened with alcohol, bruised and expressed, and treated further with alcohol till everything soluble is exhausted. We next add from 10 to 30 grains of tartaric acid, place the mixture in a flask, and heat to 170° Fahr. When quite cool, filter, wash with strong alcohol, adding the washings to the filtrate, and place the whole to evaporate under the exhausted receiver of an air-pump. If, as the liquid concentrates, fatty or other insoluble matters appear, filter again, and let the filtrate and washings evaporate under the air-pump, or under a glass bell over sulphuric acid, till nearly dry. The residue is treated with cold anhydrous alcohol, exhausting thoroughly; evaporate the clear liquid in the open air at common temperatures, dissolve the acid residue in the smallest possible quantity of water, place the solution in a small test-tube, and add little by little pure bicarbonate of soda in fine powder till effervescence ceases. Agitate the whole with five times its bulk of pure ether, and leave it to settle. When the ether is perfectly clear decant some of it into a capsule, and leave it in a dry place to spontaneous evaporation.

First Case.—The base is *volatile and liquid*. On evaporating the ether, there remain in the inside of the capsule some small liquid striæ, which fall to the bottom of the vessel. In this case, under the influence

of the heat of the hand, the contents of the capsule exhale an odour more or less disagreeable and pungent, something like that of ammonia coupled with an animal odour. We add then to the contents of the vessel from which a small quantity of ether has been decanted, two or three fluid drachms of a strong solution of caustic soda or potash, and agitate the mixture. After a sufficient time we draw off the ether into a test-tube, exhaust the mixture by two or three treatments with ether, and unite all the ethereal fluids. We next pour into this ether, holding the alkaloid in solution, one or two drachms of water, containing $\frac{1}{5}$ th part of its weight of pure sulphuric acid, agitate it for some time, leave it to settle, pour off the ether floating at top, and wash the acid liquid at bottom with fresh ether. The sulphates of nicotine, aniline, quinoline, picoline, and petinine, being insoluble in ether, will, if present, be found in the acid liquid in a pure state. Sulphate of conicine being soluble in ether, will be found partly in the ether, and partly in the acid liquid. To extract the alkaloids from the acid liquid, we add to the latter a concentrate aqueous solution of potash or soda, and agitate, and exhaust the mixture with pure ether, which dissolves any ammonia present, and the alkaloid is now free. We expose the ethereal solution to spontaneous evaporation at the lowest possible temperature; almost all the ammonia is evolved with the ether, while the alkaloid remains as residue. To expel the last traces of ammonia, the capsule is placed for a few minutes in a vacuum over sulphuric acid, and the alkaloid thus obtained in a state of

absolute purity, fit for the application of special reagents.

Second case, the alkaloid is fixed and solid.

It may happen that the evaporation of the ether resulting from the treatment of the acid matter to which we have added bicarbonate of soda, may, or may not, leave a residue containing an alkaloid. If it does, add a solution of caustic soda or potash to the liquid, and shake it briskly with ether, which dissolves the alkaloid, and it now remains free in the solution. In either case we exhaust the matter with ether. Whatever be the agent which has set the alkaloid free, whether it be bicarbonate of soda, or caustic soda, or potash, it remains on the evaporation of the capsule as a solid body, or as a milky liquid holding solid matter in suspension. The odour of the substance is disagreeable, but not pungent. It turns litmus paper permanently blue.

When a solid alkaloid is discovered, it must be obtained in a crystalline form. Put some drops of alcohol in the capsule containing the substance, decant off the clear solution, and let the alcohol evaporate spontaneously. If the alkaloid is not pure enough to crystallize, pour into the capsule some drops of water faintly acidulated with sulphuric acid, and move it about so as to bring it in contact with the substance. Generally we observe that the acid water does not moisten the sides of the capsule. The contents separate into two parts—greasy matter, which remains on the sides of the vessel; and alkaloid, which dissolves and forms an acid sulphate. We cautiously

decant the acid liquid (which ought to be limpid and colourless); the capsule is well washed with some drops of acidulated water, which is added to the first liquid, and the whole evaporated to three-fourths under a bell over sulphuric acid. We put into the residue a very strong solution of pure carbonate of potash, and treat the whole with absolute alcohol. This leaves untouched the salts of potash, but takes up the alkaloid. The alcoholic solution on careful evaporation leaves the alkaloid in crystals.

In this manner morphia, strychnia, brucia, veratria, emetina, colchicina, aconita, atropia, and hyoscyamia.

Herepath recommends for the detection of alkaloids in an unknown mixture (*e. g.* the contents of a stomach in cases of poisoning) to add an excess of magnesia and distil slowly, when volatile alkaloids, if present, will come over, and may be separated from water, ammonia, &c., by agitation with a little chloroform. By this operation aniline, nicotine, picoline, conicine, secaline, hyoscyamine, and sparteine may be isolated for further examination.

If no volatile alkaloid be detected, the contents of the retort are evaporated to dryness in a water-bath, and the residue acted on with boiling alcohol. The clear liquid is evaporated to dryness, and the alkaloid is freed from fatty matter by treatment with water acidulated with sulphuric acid, which leaves the latter undissolved. The acid solution is mixed with a slight excess of alkali, and shaken with chloroform, ether, or pure fusel-oil, on evaporating which the alkaloid is obtained in a state of purity,

Graham and Hoffman, for detecting an alkaloid (strychnia) in a mixed organic fluid, proceed thus:—A portion of animal charcoal is diffused through the liquid, and allowed to digest, with frequent stirring, from eight to twelve hours. The liquid is then filtered, the whole of the charcoal boiled with four or five times its weight of alcohol, and the clear liquid filtered off.

The alcohol may now be allowed to evaporate, and the residual alkaloid purified by means of acidulated water, followed by potash and chloroform as above.

Subjoined are the more characteristic reactions of the better known alkaloids. Concerning the different reagents employed, it must be remarked that "Schulze's liquid" consists of perchloride of antimony added drop by drop to phosphoric acid. The nitric and sulphuric acids used should be respectively very concentrated. The perchloride of iron should be as neutral as possible. In testing with the chlorides of mercury, gold, and platinum, a little muriatic acid is often requisite to be added to the solution of the alkaloid. "Otto's test" consists in moistening the substance to be tested with concentrated sulphuric acid, and then adding a small quantity of bichromate of potash, ferridcyanide of potassium, peroxide of lead, peroxide of manganese or chlorate of potash, when a series of colours will appear.

In applying these tests very small portions should be operated upon. The drops of solution, &c., may be taken up with glass rods drawn out to about

$\frac{1}{20}$ inch in diameter, and the result must be observed with a good lens, or, if need be, under a compound achromatic microscope.

The dilatation of the pupil of the eye is best observed by dissolving one grain of the substance in about sixty of water, and applying a single drop to the *conjunctiva* of the eye. The effect follows in from five to fifteen minutes. Alcoholic solutions are too strong, and are apt to produce severe inflammation of the eye.

a. Volatile bases, liquid or paste-like, giving off a pungent ammonia-like odour when warmed along with caustic potash or soda.

1. *Aniline* gives a splendid colour with solution of bleaching powder, and with Otto's test; turns litmus paper blue, and neutralizes acids.

2. *Conicine* (smell of mice); colourless oily liquid; strongly alkaline. When forming the $\frac{1}{250}$ part of a liquid it gives opalescence with Schulze's test; with nitric acid a topaz yellow. Sulphuric acid, no change in the cold; on continued heating it becomes greenish-brown, blood-red, and black; muriatic acid, a violet; weak tincture of iodine, a white precipitate; tannic acid, a white precipitate; chloride of mercury, a white precipitate; chloride of gold and chloride of platinum, yellow precipitates; acetate of lead, no precipitate; perchloride of iron, a yellow precipitate; acetate of copper, a blue gelatinous precipitate; and chloride of zinc, a white gelatinous precipitate, both soluble in an excess of conicine.

When pure conicine is carefully heated in a cap-

sule, it forms white vapours, having a strong smell of celery, mixed with that of the urine of mice.

3. *Nicotine* (smell of tobacco); oily liquid, alkaline reaction. At $\frac{1}{250}$ turbidity with Schulze's test; with strong sulphuric acid, in the cold, a wine red colour; blackens on boiling; heated with muriatic acid, a violet colour; chloride of mercury, a white precipitate; chloride of platinum, a canary-yellow precipitate, soluble in water; chloride of gold, an orange precipitate, very soluble in an excess of nicotine; tannic acid, a white precipitate; acetate of lead, a white precipitate; solution of iodine, a yellow precipitate; perchloride of iron, an ochre yellow precipitate.

4. *Hyoscyamine* (smell of mice); volatile, but may be obtained in crystals; alkaline reaction; gives a white precipitate with tannic acid, a yellowish-white with chloride of gold, and no precipitate with chloride of platinum.

5. Fixed, crystalline, or amorphous bases, giving off no pungent odour.

1. *Aconita*, strongly alkaline, neutralizes acids, but its salts do not readily crystallize; ammonia and potash give flocky precipitates, sparingly soluble in excess; carbonate of ammonia, bicarbonate and phosphate of soda, no precipitate; chloride of gold, a dense yellowish precipitate, very sparingly soluble in muriatic acid; chloride of platinum, no action; chloride of mercury, a white curdy precipitate, soluble in muriatic acid and in sal-ammoniac; sulpho-cyanide of potassium, a white curdy precipitate; tincture of iodine, a reddish-brown precipitate; tannic acid, with

one drop of muriatic acid, a flocky precipitate, which require a large quantity of muriatic acid for re-solution; picric acid, a dense sulphur-yellow precipitate, insoluble in ammonia. It dilates the pupil.

2. *Atropia*; strongly alkaline; potash, ammonia, and carbonate of potash give powdery precipitates in very strong solutions very soluble in excess; carbonate of ammonia, bicarbonate and phosphate of soda, no precipitates; chloride of gold, a crystalline, sulphur-coloured precipitate, sparingly soluble in muriatic acid; chloride of platinum, a powdery precipitate, which cakes together, very soluble in muriatic acid; sodio-chloride of iridium, no precipitate; chloride of mercury, a precipitate in very concentrated solutions; potassio-iodide of mercury, a very heavy, white, curdy precipitate; iodide and sulphocyanide of potassium, no precipitate; tincture of iodine, kermes brown; iodic acid, cold, no colour. Tannic acid, a dense flocky precipitate, along with a little muriatic acid, in an excess of which it dissolves. Picric acid, a sulphur-yellow powdery precipitate; nitric acid, no change either alone or on adding protochloride of tin. With Schulze's liquid at $\frac{1}{1000}$ it gives a white curdy precipitate; with Otto's test it gives, after some minutes, a nickel-green, then greenish-yellow, then dirty yellow, finally a yellowish resinous precipitate soluble in alcohol. Cyanogen gas gives a blood-red colour in the alcoholic solution. It dilates the pupil.

Berberine, doubtfully alkaline, gives yellow amorphous precipitates with chlorides of platinum, and mercury, tannic acid, chromate, and chlorate of potash.

Brucia, decidedly alkaline; with Schulze's liquid at $\frac{1}{1000}$ a rose-coloured precipitate, soluble while warm, re-precipitates on cooling; the supernatant liquid carmine red (best observed in solutions of the hydrochlorate); with nitric acid, a deep red solution, which changes to violet, and is discharged by protochloride of tin; with ferrocyanide of potassium (both solutions cold and saturated), a pale yellow crystalline precipitate, which, when treated with water, leaves a blue precipitate.

Caffeine, alkaline, no action with Otto's test; no action with Schulze's liquid; soluble in boiling potash; when boiled with muriatic acid and chlorate of potash, on carefully evaporating, a crystalline body is produced, the solution of which stains the skin red. The same body is produced on passing chlorine through a solution of caffeine for a short time. If spread out on a shallow dish, moistened with a very little water, and exposed to ammonia, it turns first rose red and then violet. When digested with nitric acid, and cautiously evaporated to dryness, and treated with ammonia, a red colour like that given by uric acid.

Cinchonia, alkaline; with Schulze's liquid, at $\frac{1}{1000}$ a blue flocky precipitate; with Otto's test an arsenite of copper-green, then a bright yellow-green, then a dirty dark yellow; with bleaching powder a white precipitate; with ferrocyanide of potassium, a yellow precipitate; with an alcoholic solution of ferrocyanic acid, an orange precipitate; with chloride of platinum, a crystalline, light yellow precipitate; chromate of

potash, a yellow amorphous precipitate; chloride of platinum, a pale yellow precipitate.

Codeia, alkaline; with Schulze's liquid, at $\frac{1}{1000}$ a dirty brown colour; with Otto's test, an arsenite-green, then a nickel-green, and finally a dark dirty green; with alkalies, a precipitate sparingly soluble in excess; no colour with perchloride of iron; with chloride of mercury, a white precipitate soluble in boiling water; with chloride of platinum, a pale yellow precipitate; with chloride of palladium, a yellow precipitate, which is decomposed on boiling with separation of metallic palladium; with chlorine gas, a deep brown colour; with cyanogen, a yellow, turning brown.

Daturia agrees in its reactions with atropia, and is probably identical.

Delphia, alkaline; with Otto's test, a dirty green, which then clears up, a dirty nickel-green, then a dirty yellow; with potassa a white gelatinous precipitate; with nitric acid a red, like rusty iron.

Digitaline, not alkaline; with Schulze's liquid, $\frac{1}{1000}$ a voluminous precipitate on boiling; with sulphuric acid, a beautiful purple colour, which is fugitive; with strong muriatic acid, an emerald green solution; dilates the pupil.

Morphia, alkaline; with Schulze's liquid, no action; with Otto's test, nickel-green, then copper-green, then a dark dirty green; with nitric acid, a deep red; with alkalies, precipitate soluble in excess, and in solution of sal-ammoniac; alkaline carbonates and bicarbonates, a white precipitate insoluble in excess;

sulphuric acid, no colour ; perchloride of iron, a deep blue ; iodic acid mixed with a little starch, a fine blue ; chloride of gold, a brown flocky precipitate, soluble in muriatic acid, with a green colour ; chloride of mercury a white precipitate.

Narcotina, feebly alkaline ; with Schulze's liquid, at $\frac{1}{1000}$ a copious yellow flocky precipitate ; with nitric acid, a dingy reddish yellow ; with sulphuric acid, on adding a particle of nitre, crimson ; with caustic alkalies, a precipitate not soluble in excess ; with chloride of mercury, a white precipitate ; with chlorides of iron and gold, and iodic acid and starch, no action ; with hyponitric acid, ignition, if the quantity of narcotina be at all considerable.

Papaveria, feebly alkaline ; forms salts with acids ; turns blue with concentrated sulphuric acid ; with chloride of platinum, a yellow precipitate, insoluble in boiling water and alcohol ; dissolves in muriatic acid, and on the addition of more acid, a white precipitate appears, which collects into drops, and forms an insoluble *oily* layer at the bottom of the vessel.

Phloridzine, non-alkaline ; does not form salts with acids ; gives with subacetate of lead a white precipitate ; when moistened with ammonia and exposed to the air, it acquires a blue colour, and may be dissolved in excess of ammonia. From this solution acids precipitate a red powder.

Piperia, feebly alkaline ; unites with hydrochloric acid ; gives with Schulze's liquid a yellow precipitate ; with Otto's test, a pretty green-yellow, then a nickel-green and dirty green ; when treated with nitric acid

a brown resinous body is obtained, which dissolves in potash with a splendid blood-red colour.

Quinia, alkaline; unites with acids; gives with Schulze's liquid, at $\frac{1}{1000}$ a bright yellow precipitate; with Otto's test, an arsenite of copper-green, then a beautiful yellow-green, and lastly a dark green; when dissolved in a few drops of warm acetic acid, containing a minute quantity of sulphuric acid, and the resulting solution is mixed with a drop or two of weak alcoholic solution of iodine, yields on cooling minute green crystals, which strongly polarize light (Herepath's test). When dissolved in chlorine water, and the solution subsequently mixed with a little ammonia, a fine green is produced; if ferrocyanide of potassium be added instead of ammonia, a dark red colour appears; if caustic potash is used instead of the ammonia, a sulphur-yellow colour is produced.

Salicine, non-alkaline; does not combine with acids; gives with sulphuric acid a pink colour, and with perchloride of iron a violet. Dilute muriatic acid changes it into a white insoluble powder, which in contact with strong sulphuric acid turns blood-red.

Santonine, feebly acid; dissolves in potash with a carmine colour; the crystals exposed to the sunlight turn yellow, decrepitate, and assume a golden colour; concentrated sulphuric acid dissolves it with a yellow colour, which turns red.

Strychnia, decidedly alkaline; with Schulze's liquid the solution of the nitrate at $\frac{1}{1000}$ gives a plentiful yellow curdy precipitate; at $\frac{1}{5000}$ a white flocky precipitate; at $\frac{1}{25000}$ a faint turbidity. Caustic potash gives a white

precipitate, very soluble in excess of either liquid. Tannic acid, at $\frac{1}{20000}$, gives a white curdy precipitate, very soluble in acetic acid, and if obtained from a dilute solution, in potash, forming a red liquid; bichloride of platinum, a yellow precipitate, at first amorphous, but becoming granular. In very dilute solutions it does not immediately appear.

Terchloride of gold gives a bright, yellow, amorphous precipitate, which soon becomes partly granular; a portion of the precipitate collects in minute flakes. If the precipitate obtained from very dilute solutions is boiled, it dissolves, giving a yellow liquid, from which it is again deposited on cooling. If boiled with potash it gives a fine purple liquid, with or without a purple precipitate, according to the strength of the solution. The chloride of gold gives satisfactory results up to $\frac{1}{30000}$.

Monochromate of potash gives a yellow crystalline precipitate, soluble in excess of acetic acid. At $\frac{1}{1000}$ the reaction is but sparing.

Bichromate of potash gives a brilliant yellow crystalline precipitate, less soluble in acetic acid than that given by the yellow chromate. The result is manifested up to $\frac{1}{10000}$, but not immediate.

Carbazotic acid gives a yellow pulverulent precipitate, which becomes tuft-like; at $\frac{1}{20000}$ granules are produced in a few minutes.

Chloride of palladium at $\frac{1}{100}$ gives an immediate dirty white precipitate, soluble in acetic acid, but insoluble on boiling; at $\frac{1}{1000}$ an instantaneous yellow precipitate.

Ioduretted iodide of potassium (three grains iodide

of potassium, dissolved in one fluid drachm of water, and one grain of iodine added) gives at $\frac{1}{1000}$ a copious brownish-yellow, amorphous precipitate, soluble in alcohol, ether, and in a large excess of acetic acid.

The precipitate partially dissolves in potash, but is immediately replaced by a dirty white precipitate. At $\frac{1}{80000}$ little yellow flakes.

Bromine (hydrobromic acid saturated with bromine) gives at $\frac{1}{1000}$ an immediate bright yellow, amorphous precipitate; at $\frac{1}{10000}$ a greenish-yellow precipitate; and at $\frac{1}{50000}$ a dirty yellow precipitate, which after a time redissolves.

Otto's test (the substance moistened with sulphuric acid, and a particle of bichromate of potash added) gives a dark purple. (In this manner it is not interfered with by sugar, quinia, cinchonia, starch, or dextrine.) If pure chromic acid is used the violet colour is more distinct; and strychnia may thus be detected when mixed with santonine and brucia. But morphia and its acetate mask the violet colour, sugar conceals it, and quinia turns it a pale rose colour.

Marchand triturates the substance with a little peroxide of lead and concentrated sulphuric acid, to which one per cent. of nitric acid has been added. The colour is first blue, then purple, then red, and lastly, after some hours, a delicate yellow.

Another modification of the test is to mix a few drops of pure sulphuric acid with an equal bulk of water, and allow it to cool. Chlorate of potash in fine powder is then added, and the suspected substance introduced. If strychnia be present an intense

maroon-red colour appears, gradually fading into a rose-violet. E. W. Davy moistens the suspected substance with concentrated sulphuric acid, adds a little ferridcyanide of potassium (red prussiate) in fine powder, and mixes them well together. The usual violet colour appears. $\frac{1}{5000}$ is about the limit of the colour-test, the substance being preferably dry.

Veratria, alkaline, combines with acids; with Schulze's liquid at $\frac{1}{1000}$ a dirty white flocky precipitate; at $\frac{1}{5000}$ the liquid becomes opalescent; with Otto's test, a dirty green, then a bottle green, a dirty nickel-green, which then brightens; finally a dark dirty yellow.

§ 109. OPIUM.

The composition of genuine opium varies considerably, according to the locality where it has been produced. A pure sample may contain—

Morphia.	Papaveria.
Codeia.	Meconina.
Narcotina.	Narceina.
Methylo-Narcotina.	Porphyroxine, or opine.
Ethylo-Narcotina (Narcotine of Blyth).	Meconic acid
Propylo-Narcotina	Lignine.
Opiania (<i>Engler</i>).	Bassorine.
Paramorphia, or thebaine.	Gum.
Pseudomorphia (<i>Pelletier</i>).	Caoutchouc.
	Resin.

Besides a fatty oil, an extractive acid, and a volatile poisonous principle not thoroughly understood. Some of these bodies are only occasionally present, and some are possibly not pre-existent in the plant, but

are generated by the treatment to which the opium is submitted.

Opium is adulterated with clay, sand, gums, water, starch, liquorice, and a variety of vegetable extracts. Sometimes parcels are met with from which the morphia, the most valuable constituent, had been removed.

The methods for the examination of opium turn not upon the detection of adulteration, but upon an estimation of the amount of morphia.

Half an ounce of the sample is cut in fragments, and bruised in a mortar with alcohol at 71° . The fluid is strained through lawn, the residue washed with more alcohol, the alcoholic tinctures are mixed, and filtered into a wide-mouthed bottle, and mixed with sixty grains liquid ammonia. In twelve hours the morphia, with some narcotina and meconate of ammonia, will be found deposited. The latter is removed by washing with cold water, and the crystals are then shaken in chloroform, which dissolves the narcotina, leaving the morphia untouched; or—

900 grains of opium are treated with four times their weight of boiling alcohol at 71° . The solution is decanted while hot, the grounds strained when cool, and again treated with 1400 grains of alcohol of the same strength. All these liquids are mixed, and set aside for a day in a well-stoppered phial. The liquid is then poured off from the crystals of narcotina, put into a wide-mouthed flask, and mixed with sixty grains of ammonia. A considerable quantity of morphia is thus obtained and separated from the liquor. A

little water is next added to the alcoholic liquor, and the flask is kept for two days at 75° Fahr., when a further quantity of morphia is obtained.

The amount of morphia in opium ranges from three per cent. (Egyptian) to seven (Smyrna). Instances have occurred where 10, 12, and even 13.5 per cent. have been obtained.

To detect small quantities of opium, add to the suspected substance some potash, and then shake it with ether. A strip of white blotting paper is then moistened with the liquid, dried, and again moistened repeatedly. When dry the paper is to be moistened with muriatic acid, and exposed to a current of steam from the spout of a kettle. If opium be present the paper will be coloured red.

The specific gravity of opium is about 1.336.

§ 110. PERUVIAN BARKS ; CINCHONA BARKS.

The commercial value of a sample of bark depends upon the amount of quinia (quinine) it contains. For this purpose, take 100 grains of the bark, put it in a porcelain basin, adding enough hot water to moisten the powder, and then leave it to swell. After some minutes, add 100 grains of lime (caustic) in fine powder and enough water to form a paste, which must be well mixed by stirring with a glass pestle. The whole is now perfectly dried on the water-bath, and the quinine dissolved out by a known quantity of ether, which must be completely free from alcohol and water. The ethereal solution is now placed in a stoppered flask. Now take a dilute pure sulphuric acid, of specific gravity

1.014 or thereabouts, ascertaining the exact amount of actual acid it contains, as in II. § 3. Of this standard acid, which should be kept in readiness, add a known quantity, by means of a pourette, to the ethereal solution in the flask. It is scarcely needful to say that the acid must be added in excess. Shake the acid and ether well together, and then, by means of a standard solution of pure ammonia, determine how much acid remains free. As 162 grains of quinine require for saturation forty grains of real sulphuric acid (SO^3), it is easy to calculate the per centage of quinine in the sample. To shorten the calculation, the standard solution of ammonia may be made so strong that one degree shall exactly neutralize one degree of the dilute acid.

Cinchonia is too sparingly soluble in ether to materially vitiate the result.

Quinine is rarely present to the amount of three per cent.; or—

Exhaust two grains of bark with eight ounces water, to which twenty grains of dilute sulphuric acid have been added; keep at about 130° Fahr. for twenty hours with frequent stirring, and filter. Treat the powder again twice in the same manner with eight to nine ounces water acidulated with a few drops of dilute sulphuric acid, until the last filtrate is only rendered faintly turbid with ammonia. Mix the extracts, and precipitate until a filtered portion is no longer rendered turbid by ammonia. The precipitate is then filtered, washed, pressed, dried, and weighed.

This test serves to distinguish the species of *Cin-*

chona from which a sample of powdered bark has been prepared. Yellow bark exhausted with hot water, acidulated with a little sulphuric acid added to it, furnishes a pale yellow liquid, which with excess of ammonia gives a plentiful red-brown precipitate, the liquid at the same time turning a beautiful red. Red bark gives an ochre-yellow precipitate and a yellow-brown liquid. Pale Carthagena bark furnishes a chocolate-brown precipitate and a darker yellow filtrate. Huamalies bark furnishes a dark yellow infusion, an ochreous precipitate, and a red filtrate. The false barks (*Surinamensis* and *Braziliensis*) yield with acidulated water much darker infusions, and yield no precipitate of an alkaloid with ammonia until the liquid has a very decided basic reaction.

To determine the entire alkaloids present in a sample of bark, the following process may be employed:—

a. Examination of Grey Barks.—600 grains of the sample powdered and sifted are moistened with a sufficient quantity of water acidulated with hydrochloric acid (20 parts acid to 1000 of water) and placed in a cylindrical tube; a piece of filter-paper is placed beneath, and the powder washed with the acidulated water. When the liquid passes through colourless and free from bitterness—which should occur when from 3000 to 3750 grains of liquid have passed through—no more is added. To the clear liquid add 75 to 90 grains of caustic potash and 225 grains of chloroform. The whole is well shaken for a few minutes and set aside. In about half an hour the chloroform is deposited with the cinchonine.

The clear red liquid is decanted off, the deposit washed with water by decantation, and put in a porcelain capsule. This is heated in the water-bath to expel chloroform, and the residue treated with water acidulated with hydrochloric acid. Filter, and add to the filtrate ammonia, diluted with fifteen times its bulk of water, drop by drop, stirring well each time. As soon as a white cloud appears, which no longer dissolves on stirring, no further addition is made. The reddish-brown precipitate (cinchona-red) is filtered off and washed with a little distilled water, the washings added to the filtrate, which should be colourless, and an excess of ammonia added. The precipitate is collected, dried, and weighed.

b. Examination of Yellow and Red Barks.—300 grains of bark are sufficient; 3000 grains of liquid are obtained, to which we add 75 to 90 grains of potash, and 150 grains of chloroform. The mixture is well shaken and set aside. A heavy, whitish precipitate falls; the red supernatant liquid is carefully decanted off, the residue washed and collected in a small capsule; the chloroform is allowed to evaporate spontaneously, and the residue weighed.

§ 111. QUININE AND ITS SALTS.

The various commercial salts of quinine are often found contaminated with cinchonia, quinidine, salicine, phloridzine, stearine, mannite, sugar, gum, starch, chalk, carbonate and sulphate of baryta, boracic acid, &c., besides containing an undue amount of water.

1. Dry a weighed sample very carefully at 212° Fahr. The loss = *water*.

2. Incinerate a weighed portion in a platinum crucible and weigh the residue. If the sample be free from mineral impurities nothing will remain.

In case of double salts—*e. g.*, double citrate of quinine and iron—oxide of iron will of course remain. This is taken up by digestion in muriatic acid; when a white insoluble residue points to *sulphate of baryta*. The solution of the iron is next examined by the ordinary methods for salts of *lime*, the alkalies, &c.

3. Treat a small portion with concentrated sulphuric acid. If it turns red, *salicine* is present.

4. Put a little into a porcelain capsule and add a few drops of nitric acid. If the mixture becomes first yellow, then green, and lastly, dark brown, *phloridzine* is present.

5. Digest at a gentle heat in alcohol at 90 per cent. In addition to the mineral impurities, *gum*, *starch*, *sugar*, *milk-sugar*, and *mannite*, if present, will remain insoluble. *Boracic acid*, however, may be taken up by the alcohol, wherefore a part of the solution is evaporated to dryness and then ignited in a platinum crucible, when if present, it will remain.

6. Treat a portion at a gentle heat with water to which a little sulphuric acid has been added, *stearine* and other *fats*, and *resinous matter*, if present, will separate on cooling. The sample, if pure, should dissolve without residue.

7. Put 25 grains of the sample in a flask with 150 grains of ammonia; shake well, and allow the mixture

to stand for twenty-four hours. Heat in a water-bath to expel excess of ammonia; cool, and add 240 grains of pure ether, shaking briskly. *Cinchonia*, if present, remains floating as a white insoluble layer between the upper solution of quinine in ether and the lower stratum, water containing sulphate of ammonia.

8. *Ammoniacal salts* may be detected by rubbing the sample with a little caustic soda or potash, when an ammoniacal odour will be given off.

§ 112. MUSK.

A genuine musk-pod has two small openings; it is roundish, and varies in size from one to two and a half inches in diameter. A portion of a hair taken from a genuine pod and examined with a magnifying power of 300 diameters, will be found full of regular colour cells, which are absent in the hairs of spurious pods.

The granules within should be of a very deep brown, with a faint reddish tint; they vary in size from a pin's head to a small pea, and are globular, ovoid, or irregular.

Musk should dissolve in boiling water, not leaving more than 25 per cent. of residue. The boiling solution should be almost entirely precipitated by acids, by acetate of lead, and solution of gall-nuts, but not by chloride of mercury. It should be soluble in ether, and should not fuse or inflame when ignited on a slip of platinum foil, but merely become charred. It should leave on incineration not more than 6 per cent. of a greyish ash.

The substances used for adulterating musk are clotted blood, asphalt, sand, lime, and promiscuous earthy rubbish.

§ 113. SCAMMONY.

Scammony is of a compact structure, its fracture has a greenish colour, and it emits a cheese-like odour. Its resin, as obtained by evaporating an alcoholic solution, is pale yellow, hard and brittle. If it has been exposed to much heat the colour is darker.

Scammony is adulterated with chalk, gum, and starchy or farinaceous matter. These substances are left behind on dissolving in alcohol, and may be weighed. Starch is, of course, also easily detected by the addition of tincture of iodine; which, in a pure scammony does not produce any blue shade.

It is also fraudulently mixed with common rosin, and with the resins of guaiacum and jalap.

To detect the two former, add sulphuric acid, which produces no immediate perceptible effect upon a genuine sample, and only after the lapse of some minutes does a faint colour of lees of wine appear. If, however, common rosin (colophony) or guaiacum be present, an intense red is immediately developed; which, in the case of guaiacum, turns green on the addition of water.

Nitric acid turns an alcoholic solution of guaiacum green, which passes into brown. It produces no discolouration in pure scammony.

Chloride of soda gives a beautiful green, and nitrate of silver and sesquichloride of iron a blue, in solutions

of guaiacum; whilst in solutions of pure scammony they produce no effect at all.

If resin of jalap is present, it may be detected by the action of ether, which leaves it undissolved, but takes up the resin of scammony.

Resin of scammony is completely soluble in ammonia at 0.910 specific gravity; the solution has a greenish colour.

The amount of resin of scammony in a first-rate genuine sample is about 84 per cent.; whilst in some inferior kinds it is only 6.

§ 114. GUAIAACUM.

1. To ascertain the genuineness of guaiacum wood, put a few of the shavings into a test-tube, pour over them a few drops of a solution of perchloride of mercury, and warm slightly. A bluish-green colour is at once produced with all genuine samples.—*Schwacke.*

2. The resin of guaiacum takes a greenish-blue colour in contact with chloride of lime or chloride of soda. By this reaction $\frac{1}{320}$ of guaiacum can be detected if mixed with scammony or jalap-resins.

3. The presence of manchineel in guaiacum may be detected by dissolving the sample in proof spirit, and adding a few drops of hyponitrous ether. If the solution be now diluted with water, the resin of guaiacum is precipitated whilst manchineel floats.

§ 115. RHUBARB.

To distinguish Turkey-rhubarb from the common

native species, take a little of the root, let fall on it two or three drops of the essential oil of bergamot, aniseed, or fennel ; then add a little magnesia, and rub them together for a few minutes. If the sample consist of genuine oriental rhubarb the yellow colour will remain unaltered, but if any portion of the common species be present, the shade will vary from a salmon to a full rose colour according to the quantity of the latter.

§ 116. CALOMEL (*Subchloride of Mercury, formerly considered as Protochloride of Mercury*).

Pure calomel should be entirely volatilized by heat; if any residue is found, it may consist of sulphate of baryta, sulphate of lead, chalk, carbonate of baryta, white lead, or gypsum. After accurately weighing the residue, it is treated with a little pure acetic acid. If any effervescence arises, the acid is allowed to act as long as anything is taken up; the residue is filtered off, washed, dried, &c., and weighed. Through the filtrate and washings pass a current of SH; if any blackening appears, lead is present; the current is then continued as long as any precipitate continues to be formed; the precipitate filtered off, and treated as in I. § 73. The lead found in this solution must have been present as carbonate, and consequently 104 parts of lead found in this solution represent 129 parts of white lead (sesquicarbonate) in the calomel. In the acetic solution, after removal of the lead, lime and baryta, if present, are separated and determined as in I. § 11. The residue, insoluble in acetic acid, is treated with 500 parts of water, which will take up

gypsum if present. Its amount may be approximately determined by weighing the dried residue after treatment with water, and reckoning the loss as gypsum. As, however, the sulphate of lead is not absolutely insoluble in water, this method is not perfectly accurate; and therefore where great nicety is required, a current of SH should be passed through the aqueous liquid, and any lead thrown down as sulphide is filtered off, &c., and its weight deducted from the amount calculated above as gypsum, and added to the sulphate of lead as found below. The residue, insoluble in water, may contain sulphate of lead and sulphate of baryta. The former may be removed by digestion in hot concentrated nitric acid, or in tartrate of ammonia with excess of ammonia; the insoluble matter will be sulphate of baryta. The separation may also be effected by digestion in a strong solution of hyposulphite of soda at a temperature not exceeding 68° Fahr. The sulphate of lead dissolves.

For most purposes, however, it is sufficient to weigh the total matter remaining after the calomel has been volatilized by heat.

If *common salt* (chloride of sodium) is present, the greater part of it will remain after sublimation. To separate it from other fixed impurities, the residue is digested at 161° Fahr. in dilute alcohol of 0.876 specific gravity. The salt is dissolved, whilst the other impurities mentioned above remain.

Another class of impurities cannot be detected by sublimation. Such are *metallic mercury*, which gives the sample a greyish appearance. The globules are

visible on examination with the microscope. *Sub-nitrates of mercury*; a portion of the sample heated in a small test-tube gives off ruddy fumes; or—wash the sample in cold water, evaporate the washings down to a small bulk, and test for nitric acid (II. § 5, and 3, 2). *Chloride of mercury* (corrosive sublimate) is dissolved out in alcohol, in which it may be detected by a current of SH, which turns it brown (see also II. § 160). If *sal-ammoniac* is present it may be ascertained by extracting in cold water, concentrating the solution, and adding some caustic soda or potash. On heating, ammoniacal fumes are given off, and may be made manifest by holding a glass rod moistened with muriatic acid over the capsule or test-tube, when a dense white cloud will appear. *Gums, starches*, and other organic impurities give an empyreumatic smell when the sample is heated, and may be extracted out with water.

Of the foreign substances found in calomel, metallic mercury, nitrates of mercury, and corrosive sublimate, are accidental impurities arising from neglect in the manufacture; whilst the sulphates and carbonates of lead, lime, and baryta, common salt, sal-ammoniac, and organic matter, are wilful adulterations.

To sum up, calomel should contain no matter soluble in alcohol or cold water, and nothing which does not volatilize when heated.

§ 117. CORROSIVE SUBLIMATE (*Chloride of Mercury, formerly Bichloride and Oxymuriate of Mercury*).

The chloride of mercury is generally sold in a state

of purity. It should be perfectly volatile on the application of heat, and dissolve without residue in water, in alcohol, and in ether.

§ 118. BLUE PILL MASS.

This substance, which is manufactured on a large scale for exportation, should contain 33·3 per cent. of mercury, the remainder being conserve of roses. The adulterated article contains—

Mercury,	7·5
Clay,	27·0
Prussian blue,	1·5
Sand,	2·0
Soluble saccharine matter,	34·0
Insoluble organic matter,	12·0
Water,	16·0

100·0—*Reid.*

This and similar qualities may be very easily examined by ignition. A genuine sample should not leave more than one per cent. of ash as residue. The amount of moisture is determined by careful drying as long as any loss of weight is ascertained. The soluble saccharine matter is taken up with water. The presence of prussian blue (used to colour inferior samples) is detected by rubbing up a little of the sample with a concentrated solution of caustic potash or soda, or soda-ash. If prussian blue be present, the mass will take a brownish colour. The amount of prussian blue may be ascertained by dissolving it out with a little dilute solution of oxalic acid. Nitric acid then takes up the mercury, and the sand and clay are

left insoluble. For the determination of the mercury see II. § 160.

§ 119. OXIDE OF MERCURY (*Red Precipitate*).

The chief adulterations to which oxide of mercury is liable are red lead, peroxide of iron, and brick-dust. All these remain behind if a small portion is heated to redness in a test-tube or a porcelain capsule.

Traces of nitrate of mercury sometimes remain if the sample has not been carefully prepared. To detect these, wash with cold water, concentrate the washings, and test for nitric acid (II. § 5, 3).

§ 120. WHITE PRECIPITATE (*Amido-chloride of Mercury, and Amido-chloride of Mercury with Sal-ammoniac*).

These two substances were until recently considered identical. They may be most conveniently examined by submitting them to a subliming heat, when nothing should remain.

§ 121. COPAL.

Two sorts of copal occur in commerce—the Indian and the West Indian. The former is generally in small, irregular, rounded pieces, with a finely-warted surface resembling the skin of a goose. It is of a deeper colour than the West Indian. The latter species has not a warty surface, is very pale, or even colourless, and occurs in large irregular fragments, partly with a rounded surface and partly with a shelly fracture. Copal is without scent or taste, and has a specific gravity varying from 1.04 to 1.13.

These two species may be distinguished by means of the following liquid:—Mix 60 parts alcohol at 0·813, 10 parts ether, and 40 parts oil of turpentine. In this West Indian copal dissolves, even in the cold, without any previous gelatinous swelling; whilst that of India forms gelatinous lumps, but does not dissolve.

Copal is sometimes adulterated with dammarra, cow-dee, and anime, all of which dissolve in alcohol, which copal does not.

§ 122. BALSAM OF COPAIBA (*Capivi*).

Copaiba is produced by several species of trees of the genus *Copaifera*, which flourish in Brazil, Guiana, Venezuela, and the West Indies, the chief supply being derived from *C. multijuga*, *C. officinalis*, and *C. martii*. The best quality comes from the Brazilian province of Para.

It varies much in quality according to the species, the age of the tree, the locality, and the time and mode of collection.

It is extensively adulterated with castor-oil and with crude turpentine. The best methods of detecting the impurity are:—

Boil the sample along with water for a considerable time, to expel the volatile oil. If the sample be pure, it leaves a resin, which gets dry on cooling, whereas, if castor-oil be present, the residue will remain soft. Or, put a few drops of the suspected sample on a sheet of paper, and hold it before a fire to volatilize the essential oil. If the copaiba be pure, the paper will exhibit a homogeneous and translucent stain. If castor-

oil be present, the stain will appear surrounded with a greasy border. Or, rub up one part of the sample with four parts carbonate of magnesia, and set the mixture aside. In the course of a few hours it will be transparent if pure, but turbid if castor-oil be present. Turpentine renders the balsam more tenacious, so as to cling to the sides of the bottle. It may sometimes be detected by the smell.

§ 123. GUM AMMONIACUM.

This gum, or rather resin, may be distinguished from other resins and gum resins by the blood-red colour which it assumes in contact with a solution of the hypochlorite of lime or of soda (bleaching powder). The reaction is available with the crude substance, with the pure resin, the alcoholic tincture, and serves to detect gum ammoniacum in pharmaceutical preparations.

In this manner ammoniacum may be distinguished from galbanum, sagapenum, opoponax, assafoetida, myrrh, olibanum, mastic, sandarach, resin of jalap, scammony, elemi, liquid styrax, calamite styrax, benzoin, balsam of tolu, dragon's blood, euphorbia, colophony, resin, pitch, white pitch, and turpentine.

The alkali present in chloride of soda gives with aloes and rhubarb a vinous red, which, however, need not be confounded with the blood-red hue given by ammoniacum.

§ 124. JALAP.

Jalap is adulterated with resin of guaiacum, a mix-

ture of which with jalap sometimes goes by the name of Marseilles jalap. If the sample is mixed with fifteen parts of pure water, in which have been dissolved two parts of refined sugar, and one part of clear white gum arabic, if the sample be pure, the mixture, after a few minutes standing, acquires a yellow colour. If any guaiacum be present, the colour will be greenish, verging more to blue as the proportion of guaiacum is greater.

§ 125. PETROLEUM.

Petroleum is sometimes adulterated with oil of turpentine. The sample should be rubbed up with iodide of potassium and water, which will turn yellow if turpentine is present.

§ 126. VALERIANIC ACID, SALTS OF

The valerianates are sometimes adulterated with the corresponding salts of butyric acid, and occasionally the latter are altogether substituted for the former.

Free butyric acid, as obtained by distilling a mixture of its alkaline salts with sulphuric acid, give a bluish-white precipitate with a strong solution of acetate of copper. If the valerianates be similarly treated, there is no immediate action till the mixture is shaken, when greenish oily drops fall. The true valerianates, when heated, fuse, give off valerianic acid, and at last burn with a luminous flame, and an offensive smell, all which results are wanting in the spurious samples.

Genuine valerianates of iron and quinine are solu-

ble in spirits of wine, whilst spurious samples are insoluble.

§ 127. PLATE POWDERS (*Polishing Powders*).

Plate and polishing powders may be tested for mercury—an ingredient injurious to silver, and especially to electro-plated goods—as follows:—

A small quantity of the powder is well rubbed in a mortar, with an equal bulk of moist carbonate of soda, placed in a small bulb tube and heated to redness over a gas flame. Mercury, if present, will sublime, and form small metallic globules on the cold parts of the tube. Smaller traces may be detected by repeating this experiment in the same manner, holding within the tube, at a little distance from the heated mixture, a bit of leaf gold on the end of an iron wire. Mercury, even in very minute traces, will whiten the gold.

Extract a portion of the sample with water, filter, and examine the liquid with test-paper; if acid, nearly neutralize it with ammonia, and add chloride of calcium. A white precipitate, easily soluble in muriatic acid, shows the presence of *oxalic acid*. Heat another portion of the sample with a little caustic potash, and note if *ammoniacal* fumes are given off.

The two latter ingredients are by no means objectionable. The other common ingredients of such powders, *i.e.*, rotten-stone, whiting, jewellers' rouge (sesquioxide of iron), putty powder (peroxide of tin), &c., act mechanically by friction, not chemically by dissolving rust, &c., off the surface to be cleaned.

§ 128. MYRRH.

Myrrh is adulterated with pseudo-myrrh, *Myrrha indica*, *Bdellium indicum*, and *B. africanum*. Pseudo-myrrh consists of large pieces of different forms, the bulk of which appear to be fragments of cylindrical bodies; they are externally coated with dust, and have a dirty reddish-brown colour; the surface of fracture is tolerably even, of a vitreous lustre, brownish-yellow colour, and nearly as transparent as gum senegal. It has a faint myrrh-like odour, and a disagreeable bitter taste. In nitric acid it dissolves to a bright yellowish liquid, from which water separates small yellowish particles. Genuine myrrh gives, with nitric acid, a transparent dirty yellow liquid. *B. indicum* is not dissolved by nitric acid; it softens, becomes whitish and opaque. Filter paper, moistened with the alcoholic extract of myrrh, and then with nitric acid, acquires a blood-red colour, whilst pseudo-myrrh and bdellium show only a yellow or brownish colouring. *B. indicum* is, moreover, distinguished by its greenish-brown colour, its turpentine scent, and bitter acrid taste.

Genuine myrrh yields a bright golden yellow tincture, and an opaque whitish residue; pseudo-myrrh, a light yellow tincture, and a semi-transparent residue; *Myrrha indica*, a dark yellow tincture, and an opaque residue. In the first and last an addition of water gives a milky turbidity, but in the second no change.

If twenty drops of the tincture are mixed with six drops of nitric acid (rather dilute), genuine myrrh

gives a yellowish-white opacity, and after a time the circumference of the liquid acquires a bright violet colour, while the centre remains yellow. *M. indica* acts similarly, but the colour is darker; pseudo-myrrh does not exhibit the reaction at all.

Fuming nitric acid produces, with the tincture of genuine myrrh, an umber-brown, and finally a violet colour; on evaporation, a darkish gamboge-yellow residue appears; *M. indica* gives the same reaction; pseudo-myrrh, no change. *Bdellium indicum* and *africanum* are distinguished by their not assuming a violet colour with nitric acid. About ten grains of myrrh, shaken with an ounce of water and filtered, yield, with a solution of acetate of lead, a considerable precipitate. *Bdellium*, treated in the same manner, scarcely shows any opacity.

Genuine myrrh should dissolve completely and rapidly if powdered, left mixed with its own weight of powdered sal-ammoniac, and then treated with fifteen times its weight of water.—*Righini*.

On ignition, the residue left by myrrh should not exceed 3.5 to 3.8 per cent.; if greater, mineral impurities are present.

§ 129. AMBERGRIS (*Amber grease, Grisamber*).

Ambergris is a fatty concretion containing ambreine, which may be extracted from it by boiling alcohol. On cooling the ambreine is deposited. It is distinguished from the fatty acids by its insolubility in caustic alkali. If ambergris is pierced by a heated needle, a pleasant and peculiar odour should be evolved;

and the needle when withdrawn is found perfectly clean.

§ 130. CAMPHOR.

Camphor is generally found pure; it should be perfectly volatile when heated to 400° Fahr., and dissolve in alcohol, ether, chloroform, and nitric acid.

Artificial camphor may be distinguished from the natural kind by means of polarized light. If small fragments of each be placed on glass slips, and a drop of alcohol added to each, they dissolve, and speedily recrystallize. If the crystallization of natural camphor be watched with the microscope with polarized light, a beautiful display of coloured crystals is seen. The artificial kind offers nothing similar.

§ 131. BENZOIN (*Gum benjamin, Assa dulcis*)

AND BENZOIC ACID.

The value of a sample of benzoin is best found by ascertaining its per centage of benzoic acid. This may be performed by boiling a known weight of the sample in a solution of carbonate of soda, precipitating the solution with muriatic acid, and boiling the precipitate in water. Evaporate to dryness and weigh; or—

A known weight of the sample is digested in a flask with sulphuric acid; a larger flask is then inverted over the mouth of the former, and heat applied. The benzoic acid sublimes and condenses in the interior of the second flask, from which it is swept with a feather and weighed.

Benzoic acid should be colourless, perfectly soluble in alcohol; in boiling water, from which it is deposited in a solid mass on cooling; in strong sulphuric acid, from which it is thrown down on the addition of water; and in alkalies, from which it falls on adding muriatic acid.

It should also volatilize without residue when heated.

§ 132. ASSAFŒTIDA.

Treat part of the sample with alcohol; resin and oil are dissolved, leaving gum and impurities. The resin, freed from the alcohol by evaporation, if unmixed with other resins, is of a dirty white, which, on exposure to the air, turns rose coloured. It dissolves in strong sulphuric acid with a green colour, and water throws down rosy flakes from the solution. If a portion be ignited, the residual ash should not weigh above 4 per cent., chiefly gypsum. The specific gravity of the pure gum resin is about 1.32.

§ 133. URIC ACID.

If a fluid is under examination, albumen, if present, is removed by boiling, and filtering off the coagulated mass. The fluid is mixed with acetate of lead, filtered immediately, and the filtrate mixed with the basic acetate of lead. The whole of the uric acid is thrown down within twenty-four hours as urate of lead.

Solids (animal organs or entire animals) are well rubbed up with pounded glass, treated with two or three times their volume of alcohol, and left standing

for eighteen hours. The fluid is then pressed out and filtered; and the whole residue, which contains all the uric acid, is uniformly diffused through water, and heated in the water bath for an hour. The fluid is then separated by pressure, and the urate of lead thrown down as urate of lead. This is then decomposed by HS whilst suspended in water, and the fluid is heated to boiling, and filtered. On evaporating the filtrate, which should be colourless, the uric acid separates in colourless or slightly brownish crystals. These crystals, if dissolved in dilute nitric acid, evaporated, and treated with an excess of ammonia, acquire a beautiful purple red colour.

§ 134. DRAGON'S BLOOD; GUM-DRAGON.

The dragon's blood of commerce is very often adulterated with common rosin; some samples are even entirely made up of rosin and some red colouring matter, such as red ochre, colcothar, ground brick, red santal wood, &c.

Genuine samples dissolve in caustic potash, without alteration of the natural red colour; with sulphuric acid they take a fine yellow tint. Dragon's blood should dissolve in alcohol; and the tincture, which is blood-red, gives a brick-red precipitate with neutral acetate of lead. If common rosin is present, the potash dissolves it less easily; and sulphuric acid gives it a brownish tint. Samples containing rosin, when boiled in water, communicate to it a distinct resinous smell and taste, whilst good dragon's blood communicates to water only an earthy taste. The best

way of applying these tests is to take about fifteen grains of the sample to be examined, and treat it with 150 grains of moderately strong alcohol. A part of the alcoholic tincture is precipitated with the acetate of lead, and another portion of the tincture is evaporated to dryness, and the residue thus obtained is submitted to the action of potash and of sulphuric acid.—*Pommier*.

§ 135. HOPS.

Weigh out a portion of the sample, and digest it for some time in alcohol in a covered beaker. Pour off the liquid, wash the residue in some more alcohol, dry the residue carefully at a low temperature, and weigh. The loss should not be less than from 9 to 12 per cent. If it fall short of this the hops are deficient in lupulin, and are of inferior quality.

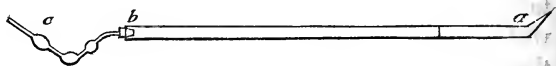
To ascertain whether hops have been sulphured, put a portion of the sample into a flask fitted up for generating gas; add a few fragments of pure zinc, and a little pure hydrochloric acid. The gas evolved is conducted into a solution of the nitro-prusside of sodium, which, if sulphur is present, will assume a beautiful, but fugitive, purple colour.

§ 136. NITROGEN AND AMMONIA.

The substance is reduced to powder in the mortar, if possible, otherwise by rasping and chopping. It is then carefully dried over sulphuric acid. Weigh out from ten to five grains, according as the sample is poor or rich in nitrogen, and mix it with an excess of

soda-lime (one part of caustic soda mixed with three or four of caustic lime), the quantity of which should be sufficient to fill half the combustion tube. This is a tube of hard German glass—fig. 16—about

Fig. 16.

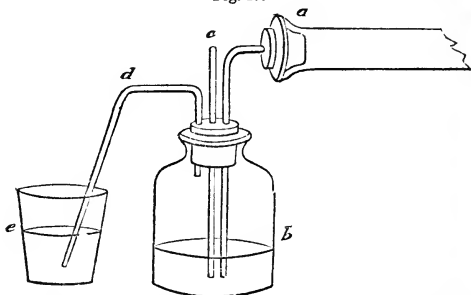


eighteen inches long, and one-third inch wide. One of its ends, *a*, is drawn out to a point and turned upwards. The substance having been mixed with the soda-lime in a warmed mortar is put into the combustion-tube, first cleaned and dried. The mortar is rinsed out with a little more soda-lime, with which the tube is filled to about an inch from the mouth. A tuft of asbestos is then introduced. The tube should be filled loosely so as to allow a free passage for the gasses. On giving it a gentle tap, a slight vacancy should appear all along the upper side from end to end. By means of a good cork, a Wills' nitrogen apparatus, *c*, is joined to the open end of the tube. It is previously charged with dilute muriatic acid (specific gravity 1.13) until its two larger bulbs are about one-third full. The combustion-tube is then placed in the channel of Hoffman's new "atmopyre" gas furnace. Open and light the jets toward the mouth of the tube, *b*, and bring that portion to dull redness. Open then the jets at the other end, *a*, and bring that portion into the same state. Then, beginning at the mouth end, open by degrees rank after rank of the jets until the entire tube is ignited.

The jets should not be turned on full at once, but gradually, and the whole conducted so that bubbles of gas may pass through the bulb-tube in a regular, but not too rapid, succession. When the current of gas has ceased, and the contents of the combustion-tube appear white, the turned-up end is broken off with a pair of forceps, and a current of air is drawn through the apparatus. The gas is then turned off, the apparatus disjoined, and the contents of the bulb-tube poured into a capsule. The nitrogen of the substance is now present in this liquid as chloride of ammonium, and its amount may be variously determined. Where very great accuracy is not needed, it may be carefully evaporated to dryness at a gentle heat in a balanced capsule. Fifty-three parts of the dry residue represent fourteen of nitrogen. Or, the bulb-tube, having been previously balanced, is weighed again after the introduction of the muriatic acid at the beginning of the analysis. The weight and strength of the acid being thus known, we may calculate what amount of ammonia would be required for its saturation. At the end of the analysis the amount of muriatic acid left unsaturated is determined by an acidimetical experiment (II. § 3), using, of course, the ammoniacal test-liquor. We thus find how much muriatic acid has combined with the ammonia generated in the process; and as seventeen parts of ammonia represent fourteen of nitrogen, we calculate the amount of the latter present in the sample. Or, instead of a bulb-tube we may use two wide-mouthed bottles—fig. 17—each containing half an ounce of abso-

lute alcohol, in which tartaric acid has been dissolved. To the end of the combustion-tube is fixed, by means of a good cork, a tube bent at right angles, *a*, passing through a hole in the cork of the first bottle, and dipping into the alcohol. A straight piece of tube, *c*,

Fig. 17.



open at both ends, passes through the same cork, and dips also into the alcohol as a safety tube. Lastly, a tube bent twice at right angles, *d*, passes with one limb through the same cork, without dipping into the liquid, while its other limb dips into the alcohol in the second bottle. When the operation is complete, the bottles are opened, the precipitate (bitartrate of ammonia) collected on a weighed filter, washed with absolute alcohol, and carefully dried at 212° Fahr.; 100 parts of this precipitate represent 8.4 of nitrogen.

Ammonia (preformed) existing free or combined in mixtures and compounds, is determined in the same manner as nitrogen. Where, however, as in guano, ammoniacal salts are found along with non-ammoniacal compounds of nitrogen, the former are

determined as follows:—Put ten to fifteen grains of the substance in a small stoppered retort, to which the bulb-tube with muriatic acid is then joined by means of a good cork. Pour into the retort some solution of caustic potash, and apply heat as long as any fumes are given off, and determine the amount of muriate of ammonia formed in the bulb-tube as above.

Nitrogen in its oxygen compounds may be thus determined:—Weigh out five to ten grains of the substance to be analyzed; and, if possible, dissolve it in a little water. Put, meantime, from 200 to 300 grains of zinc in thin clean fragments into a flask with some water. Pour one half to three-fourth ounce muriatic acid, of specific gravity 1.17, into a measuring-glass, and add about one-tenth to the zinc and water. When effervescence has commenced, add a little of the substance in question. Immerse the flask in cold water, if needed, to keep down the temperature. Add by degrees portions of the acid, and of the sample alternately, reserving about one-fourth of the acid, which is very gradually added at the last. When action has ceased, pour off the liquid from the undissolved zinc, which is washed, and the washings added to the solution. The whole is then distilled with lime or potash, and the gases received in a bulb-tube as above, and the amount of ammonia determined. An equivalent of ammonia formed, represents an equivalent of nitrogen present in an oxidized state.

For the success of this operation it is essential that the temperature be not allowed to rise during the solution of the zinc.—*Nesbitt*.

The presence of nitrogen in organic bodies may thus be detected :—A small quantity of the substance powdered, chopped, rasped, or otherwise broken up, is ignited with a small quantity of potassium. The residual mass is extracted with water, acidulated with muriatic acid, filtered and tested with a mixture of proto- and per-sulphate of iron. If a blue precipitate is formed, nitrogen was present. If no blue precipitate appears, the vessel should be set aside until the next day, when a small blue precipitate will sometimes appear, if the quantity of nitrogen be very minute.

§ 137. GUANO.

For all practical purposes guano may be analyzed as follows :—

1. Weigh out 100 grains on a counterpoised piece of filtered paper. Place the paper with its contents under the drying apparatus, and allow it to remain there until the weight no longer decreases. The loss is stated as *moisture*, and may vary from 4 to 16 per cent. If the counterpoise is of paper, it must be of the same kind as that on which the guano lies, and must be put into the drying apparatus also.

2. The dried guano, No. 1, is now put in a platinum crucible and exposed to a low red heat till all black particles have disappeared. It is now again weighed. The loss, which should be from 50 to 60 per cent., is *organic matter*. The weight of the platinum crucible should be previously exactly known.

3. Digest the ash of No. 2, which should be of a

pearly-grey colour (without any red or ochre-yellow particles) in hot water as long as anything is taken up. Filter, dry, and weigh the undissolved residue, and note the loss as *alkaline salts*; namely, sulphates and phosphates of potash or soda, and chlorides of potassium and sodium. This should be 4 to 6 per cent.

4. Treat the residue from No. 3 with hot hydrochloric acid; filter, wash, ignite, and weigh. The loss, 14 to 26 per cent., is noted as *earthy salts*; namely, phosphates of lime and magnesia, accompanied by carbonates arising from the decomposition of the oxalates.

5. The residue of the last operation is *sand*, which should not exceed $3\frac{1}{2}$ per cent.

The solutions, Nos. 3 and 4, are now submitted to special examination.

6. The former is divided into three equal parts, by means of a pourette, or graduated dropping tube.

A. In one of these determine the *sulphuric acid*, if present, as in I. § 2; or II. § 6.

B. In another, determine the *chlorine* of the alkaline salts, as in I. § 1, *b*; or II. § 4.

C. In the third, determine the *phosphoric acid* by taking a known excess of the purest iron, and dissolving it in pure hot nitric acid by degrees. The solution is poured into C, and an excess of ammonia added. The whole is digested for some time at a moderate heat, the precipitate thrown on a filter, well washed with hot water, dried, ignited, and weighed. Every grain of metallic iron employed, produces 1.44

of peroxide; and all excess of weight beyond this proportion is phosphoric acid.

D. The *potash* may be determined, if required, by collecting the filtrates from A, B, and C, adding a little sulphuric acid to remove excess of baryta, and hydrochloric acid to throw down excess of silver. Filter off the mixed precipitate, wash carefully, add the washings to the filtrate, neutralize it with ammonia, add a little oxalate of ammonia to throw down any lime which may be in solution; let the whole stand in the cold for some time, and filter off any precipitate which may appear. Evaporate the filtrate to dryness in a platinum capsule, and when dry, raise the heat to redness to drive off ammoniacal salts, and proceed as in I. § 1, &c.

It is rarely requisite in a commercial examination of guano to determine more than the phosphoric acid, C; the result being given as—alkaline salts *x*, containing *y* per cent. of phosphoric acid.

7. The acid solution, No. 4, is next divided into two parts of known bulk.

a. In one determine phosphoric acid, as in I. § 18. Every 4.6 parts of phosphoric acid found, represent ten parts of ordinary insoluble phosphate of lime present in the sample. Where time is an object, and very great exactness is not required, the solution is freed as far as possible from excess of acid by evaporation, and ammonia is carefully added as long as anything is precipitated. The precipitate (phosphate of lime) is filtered off, washed, dried, ignited, and weighed.

Tissier adds to the solution containing the phosphate of lime, boracic acid in excess, and then solution of borax, until the acid present is neutralized. Phosphate of lime is precipitated, filtered, washed, dried, ignited, and weighed.

b. If the amount of phosphate of lime be deducted from the entire weight of solid matter in No. 4, the residue will be *carbonates of lime and magnesia* (arising from decomposition of the oxalates by heat), unless sulphate of lime be also present. In this case, the joint weight of the phosphate and sulphate, as found in *c*, is deducted from the weight of earthy salts in No. 4, and the residue will be carbonate. If a considerable effervescence takes place on treating the residue, No. 3, with muriatic acid, it then appears that carbonate of lime has been, through negligence or design, added to the guano. Its amount in such cases may be directly estimated, by igniting a fresh weighed portion of guano, extracting the residue with water, and placing the part insoluble in water in the apparatus, I. § 8, *b*, and proceeding as there laid down. Every 4·4 parts of carbonic acid found, represent ten parts of carbonate of lime present in the guano.

c. To the other portion of the solution, No. 4, add chloride of barium in excess. If a precipitate appear sulphuric acid is present, which is determined in the usual manner. Every four parts of sulphuric acid found represent 6·8 of *anhydrous* sulphate of lime existing in the residue from No. 3, or 8·6 of ordinary hydrated gypsum in the original sample. Sulphate of lime is

very rarely present in a good natural guano, and then only to a small extent.

Note.—When gypsum or sulphate of lime is present in guano, its water of hydration is driven off during incineration, and makes the amount of organic matter appear too large. The latter in such cases must be corrected by deducting 5.3 for every 20 of anhydrous sulphate of lime found in the guano after ignition. The amount thus deducted is added to the sulphate of lime. Thus—Loss on ignition in a sample, 54; amount of sulphuric acid, 5; combined with lime, 3.5; making dry sulphate, 8.5. We deduct, therefore, from the organic matter 2.25, and enter the results:—

Organic matter,	51-75
Hydrated sulphate of lime,.....	10-75

It is generally sufficient in the commercial analysis of a guano to determine the phosphate of lime in No. 4, and the phosphoric acid in No. 3.

8. A fresh portion of the sample is weighed out, and burned with soda-lime in a combustion-tube in the ordinary manner, for the determination of *nitrogen* (II. § 136).

The results are tabulated as follows, *e. g.*:—

Moisture,.....	8.0
Nitrogenous organic matter,	72.0
(Containing nitrogen = 17.5 ammonia.)	
Alkaline salts,	4.8
Phosphoric acid,	
(Containing phosphoric acid = 1.1 neutral phosphate made soluble.)	
Phosphate of lime,	12.2
Carbonate of lime,.....	1.5
Sulphate of lime (if any),.....	0.6
Sand, &c.,.....	1.4
	<hr/>
	100.0

Underneath the organic matter write in parenthesis the amount of *nitrogen*, and its equivalent in ammonia.

Underneath the *phosphoric acid* found in a soluble state, write in parenthesis its equivalent in neutral phosphate of lime rendered soluble, one part of acid representing 2.1 of this salt.

The value is generally subjoined on the following principles :—

Organic matter is valued at the rate of £1 per ton ; phosphate of lime (insoluble), £8 ; phosphate made soluble, £32 ; ammonia, £64 ; sulphate of lime, £1 ; nitrogen (present as nitric acid), £100 ; alkaline salts, £1.

Potash and soluble silica are by some agricultural chemists valued at £1 per ton, whilst others rate the former at £16, and the latter at £8 per ton.

Water is considered worthless, and for carbonate of lime a deduction ought to be made.

From these values it is easy to calculate what any sample of guano, whose composition is known, is worth to the consumer. The sum thus found, or the agricultural value, ought to be higher than its market price, or it is no longer advantageous to the farmer.

A variety of characteristics aid in judging of the quality of guano. If the specific gravity is above 1.750, mineral impurities may be suspected. If the ash obtained on igniting a sample be of a yellow, red, or brown colour, the sample has probably been mixed with ferruginous marl, ochre, &c. Sawdust, a pretty frequent adulteration, is detected by weighing out 500 grains, and stirring them up in cold water. The

sawdust rises to the surface, and may be skimmed off, dried, and weighed.

Fragments of feathers may occur in genuine guano; but hairs are a proof of adulteration.

Great care is requisite in sampling guanos, &c., for analysis, so as to obtain a fair average.

In all cases where the solution of a substance has to be divided into two or more portions for the simultaneous determination of different constituents, the operator must bear this in mind in calculating and tabulating the results.

When guano is used for the manufacture of murexide, its value depends solely upon the per centage of uric acid, for the determination of which see the next section.

§ 138. GUANO (*Further Particulars*).

If a more comprehensive analysis of guano is required, the following operations may be superadded to those directed in § 137.

1. Take 200 grains of the sample and digest them in water. Filter off the solution, and dry the residue.

2. Take half the solution, place it in a small retort, to which is attached a bulb-apparatus charged with pure hydrochloric acid. Add a sufficiency of hydrate of lime to the liquid in the retort, and apply heat as long as bubbles of gas are observed to pass over. The liquid, when the operation is over, is poured out of the bulb-apparatus into a light, shallow platinum or porcelain capsule of known weight, evaporated to

dryness at 212° Fahr., and weighed as chloride of ammonium. The nitrogen thus obtained exists in the sample as soluble ammoniacal salts.

3. Divide the other half of the solution into three equal portions, A, B, and C. Acidulate A with nitric acid to expel carbonic acid, then add nitrate of iron and ammonia to remove phosphoric acid. Filter, dilute the filtrate with water, and add nitrate of lime. If any oxalic acid is present, oxalate of lime is thrown down. This precipitate is treated as usual, and the amount of oxalic acid calculated from its weight.

The sulphuric acid, chlorine (II. § 137, 5, *a* and *b*), phosphoric (do. *c*), and oxalic acids are assigned to the bases potash, soda, and ammonia, which have been found. The sulphuric acid is allotted to its equivalent of potash, any excess of the former being assigned to the soda, and of the latter to the phosphoric acid. The oxalic acid is supposed to be in combination with a part of the ammonia. Chlorine receives its equivalent of sodium, and the rest of the phosphoric acid goes to the ammonia. Should an excess of base be found, and should the aqueous solution of the guano effervesce with acids, part of the ammonia must exist in the state of carbonate. To determine this a fresh portion of guano (about 500 grains) is digested in water, the solution filtered off, concentrated, and the carbonic acid determined as in I. § 8, *b*.

The second portion of 3 B, is now evaporated to dryness in a balanced capsule, weighed, ignited, and weighed again. The difference between these two weighings shows the amount of soluble organic mat-

ter, together with ammonia, and the oxalic acid and chlorine combined therewith. The last weighing represents the salts of potash and soda, together with the phosphoric acid existing in combination with ammonia. These two results are useful in controlling the assignment of the acids to the bases.

The third portion, C, is now evaporated to dryness in the water-bath, and extracted with alcohol of 0.825 at a gentle heat, in a stout flask provided with a stopper. The alcoholic solution is decanted off, evaporated to dryness in the water-bath in a balanced capsule, and weighed. The result is *urea*.

4. The portion of No. 1, undissolved by water, is now divided into three equal parts.

A. One of these portions is digested in a large excess of borax-water (1 part borax to 100 of water), filtered, and the filter with its contents dried in a steam-bath, and weighed. The loss consists of *uric* and *ulmic acids*. To separate these two, the filtrate is precipitated with acetic acid in excess. The precipitate is filtered, dried, and weighed, as *uric acid*. The difference between this weight and the above loss is of course *ulmic acid*.

B. The second portion is treated precisely as in No. 2. The ammonia thus obtained exists in the sample in insoluble combinations; *i. e.*, urate and ultimate of ammonia, and double phosphate of ammonia and magnesia.

C. The third portion is ignited and treated with hot nitric acid, filtered, and well washed. In the filtrate, to which the washings are added, determine

phosphoric acid by means of molybdate of ammonia. In the filtrate, after removing excess of molybdenum by means of HS , determine *lime* and *magnesia* in the usual manner.

5. The residue of 4 A, remaining undissolved after the action of borax-water, is dried, ignited, and placed in the apparatus for determining carbonic acid (I. § 8, *b*). From the amount of carbonic acid thus ascertained, the quantity of oxalic acid, and consequently of oxalate of lime, is easily calculated (2.2 per cent. carbonic acid representing 3.6 per cent. oxalate of lime).

From the data thus ascertained, in addition to those obtained, (II. § 137), the exact composition of any sample of guano may be deduced. The total loss on ignition—*minus* the soluble ammoniacal salts, the oxalic acid, urea, urate and ulmate of ammonia—is entered as nitrogenous organic matter.

§ 139. BONE-ASH, PHOSPHOROLITES OR COPROLITES,
APATITE, PHOSPHORITE, BONES.

Powder, or, where that is not practicable, rasp finely, and determine moisture, and, where it exists, organic matter, as in guano. In recent bones determine the nitrogen. In all determine phosphate of lime—the constituent on which their value depends—as in guano, except iron and alumina be present, when phosphoric acid must be determined as in I. § 18. In many samples of apatite, fluorine is present as fluoride of calcium. In this case a weighed portion of the sample is digested with an excess of sulphuric acid in a platinum capsule, and heated until all fluorine and the

excess of sulphuric acid are expelled. The residue is then dissolved in muriatic acid, and in the solution phosphoric acid and sulphuric acid are determined. The amount of the latter will be proportional to the fluorine expelled—every 4 grains of sulphuric acid found representing 1.87 of fluorine. Any sulphuric acid pre-existing in the sample must be, of course, determined in another portion, and its amount deducted from the total of sulphuric acid as found above, and the amount of fluorine estimated from the remainder.

Carbonates are determined as in I. § 8, *b*. Chlorides present are estimated in a nitric acid solution.

The value of all these substances is deteriorated by the presence of any large amount of carbonates, which waste a quantity of sulphuric acid, whether the sample be employed for manure-making or in the manufacture of commercial phosphorus, &c.

Coprolites are less valuable in proportion, for agricultural purposes, than recent bones, on account of their dense structure.

§ 140. ARTIFICIAL MANURES (*Superphosphates, Blood-manures, &c.*).

These substances are frequently more complex than guano, and their perfect analysis presents many difficulties. Their analysis for commercial purposes may be effected as follows:—

1. Determine *moisture* as in guano.
2. Ignite the dried residue. The loss represents organic matter *plus* free sulphuric acid, water of hydration contained in gypsum, and sometimes also nitric acid.

3. Dissolve the residue after ignition in hydrochloric acid, filter, wash, and dry. Note the residue as *silica* (insoluble).

4. Take a fresh portion of the dried sample (fifty grains), digest thoroughly in boiling water, filter, and wash slightly. Dry and weigh the residue. The loss = amount of *soluble matter*.

5. Divide the filtrate of No. 4 into two equal portions.

a. In one of these determine the amount of phosphoric acid.

The proportion thus found represents *soluble phosphate of lime*, and is entered as such in the results; 7.2 parts of phosphoric acid in this state requiring 2.8 of lime.

b. The other portion is evaporated to dryness in a platinum capsule, and heated sufficiently to volatilize ammoniacal salts, if present. The residue is then redissolved in water acidulated with nitric acid. Any portion remaining undissolved is filtered off, &c., and weighed as *soluble silica*. In the filtrate, *chlorine* is determined in the usual manner, and accounted for as *chloride of sodium*.

6. The portion of No. 4 insoluble in water is next dissolved in hot hydrochloric acid, and the solution divided into two equal portions.

a. In one of these determine phosphoric acid (which is entered with its equivalent of lime as *insoluble phosphate*).

b. In the remaining portion determine sulphuric acid, accounting for the amount found as *sulphate of lime*.

N.B. The matter insoluble in water (No. 4), *minus* the insoluble silica (No. 3), should equal the joint weight of insoluble phosphate and sulphate of lime. If it exceed these two, the difference is probably *carbonate of lime*. To determine this, take a fresh portion of the sample, remove all soluble matter by means of water, and place the residue in the apparatus for determining carbonic acid.

7. Determine nitrogen in a portion of the original sample.

The results are then tabulated in the following manner:—

Water,	8.0
Organic and volatile matter, (containing nitrogen 8.)	32.5
Alkaline salts,.....	6.2
Soluble silica,	1.8
Soluble phosphate of lime, (= 35.9 neutral phosphate made soluble.)	23.0
Insoluble phosphates of lime and magnesia,	6.3
Sulphate of lime,.....	20.0
Carbonate of lime,	0.7
Sand, &c.,.....	1.5
	<hr/> 100.0

The valuation of artificial manures is performed in the same manner as that of guano.

§ 141. OIL CAKE, SEED CAKES, CATTLE-FEEDS.

The value of seed cakes as food for cattle depends upon their per centage of nitrogen and oily matter. As manures, their nitrogen and phosphates constitute the valuable ingredients.

1. Crumble the cake into powder, weigh out 100

grains, and place them in a very small flask. To this attach, by means of a small tube bent at right angles, a tube filled with chloride of calcium, or with asbestos moistened in concentrated sulphuric acid, and carefully weighed. To the other end of the chloride of calcium tube attach another tube bent at right angles, and dipping at its lower extremity into some sulphuric acid in a small beaker. Apply a gentle heat (not exceeding 212° Fahr.) to the flask for three or four hours, then withdraw the chloride of calcium tube, and weigh. Its increase of weight shows the amount of *moisture* in the sample.

2. Weigh out 50 grains of the crumbled sample, and treat them repeatedly with ether in a displacement apparatus, until nothing further is taken up. Place the ethereal extract in a balanced capsule, evaporate the ether away at a very gentle heat, and weigh the residue, *oil* or *fat*.

N.B. As certain oils, *e.g.*, lintseed, absorb oxygen, and thus gain weight, the following method of evaporating the ethereal solutions of such oils will be preferable:—Take an air-pump receiver, grease its edges, and fill it at the mercurial trough with pure dry hydrogen gas. Unscrew the pump-plate, set the capsule upon it, and, raising up the jar out of the mercury, immediately apply it to the plate; screw the latter into its place again, and exhaust; the ether will rapidly evaporate, and the oil will soon be obtained in a dry state. In this manner atmospheric air may be almost entirely kept from contact with the oil during evaporation.

3. Burn another portion of 100 grains to ashes.

The loss, *minus* moisture, represents *total organic* matter; the residue, total inorganic matter, which may be further examined for phosphoric acid.

The portion of the residue insoluble in acids is *sand*, which may be an intentional fraud, or a mere accidental impurity.

4. In a fresh portion of the sample determine nitrogen (I. § 136).

If sawdust is present, both the oil and the nitrogen will be deficient. Seeds of an inferior kind are best detected by the microscope. Mustard seed is frequently added to rape cake, which it renders sometimes fatal to horned cattle. To detect it, pulverize about two ounces of the cake, put it into a beaker, pour upon it about the same quantity of boiling water, cover it with a glass plate, and let it stand for some time. On uncovering the beaker it emits the very peculiar smell of a dirty mustard-pot. The odour of pure rape cake, similarly treated, although acrid, is totally distinct.

The constituents of the principal kinds of seed cake are about as follows:—

	Water.	Oil.	Organic matter.	Ash.	Phosphates	Nitrogen
CAMELINA.....	14·5	12·2	65·1	8·2	4·2	5·57
HEMP.....	13·8	6·3	69·4	10·5	7·1	6·2
RAPE.....	13·2	14·1	66·2	6·5	6·5	5·5
BEECH.....	14·0	4·0	75·8	6·2	2·1	4·5
LINTSEED.....	11·0	12·0	70·0	7·0	4·9	6·0
POPPY.....	11·0	14·2	62·3	12·5	6·3	7·0
SESAMUM.....	11·0	13·0	66·5	9·5	3·2	5·57
PISTACHIO.....	12·0	12·0	71·0	5·0	1·20	6·07

The amount of phosphoric acid is given in its equivalent of "neutral phosphate made soluble."

Compound "Cattle-feeds" may be examined as follows:—

With the microscope ascertain whether oats, barley, or maize are present. The latter is most usual, and is very easily recognized.

Extract a portion with cold water. Test for saccharine matter as follows:—Take a slip of white merino, or any woollen tissue free from cotton or linen, steep it in a concentrated solution of bichloride of tin, and dry. Dip it in a little of the filtered extract, and heat it to about 300° Fahr. If sugar is present, it will turn black.

Test another portion for tannin with a solution of gelatine.

The joint presence of sugar and tannin renders it likely that the feed contains *carrobs* or *locust beans*.

Moisture, total organic matter, total inorganic matter, and nitrogen, are estimated as in guano and seed cake. Oily and fatty matters are determined as in seed cake; common salt, as in guano or manures; tannin, as in catechu; sugar and starch, see II. §§ 90 and 91. For antimony, which may be present as tartar emetic or as sulphuret, extract a portion with nitromuriatic acid in excess, and proceed as in I. § 86.

§ 142. LIMESTONE, MAGNESIAN LIMESTONE, DOLOMITES.

Dry a weighed portion of the mineral, finely powdered, at 212° Fahr., and note the loss as *moisture*.

Dissolve in dilute nitric or hydrochloric acid, filter,

wash, dry, ignite, and weigh the residue, which is *sand and clay*.

Evaporate the filtrate to dryness, moisten with a little muriatic or nitric acid, add water, heat, filter, wash, &c., and weigh the residue as *soluble silica*.

The acid solution is now treated with a current of chlorine gas, and then ammonia (perfectly free from carbonate) is added in excess. Let the mixture stand for some time in a covered vessel at a gentle heat. Filter rapidly, avoiding access of air as much as possible. The precipitate, which may contain oxide of iron, alumina, manganese, phosphoric acid, and traces of lime and magnesia, is washed, redissolved in nitromuriatic acid, heated to expel excess of acid, and reprecipitated with ammonia with the same precautions as before. The precipitate is washed, dried, &c., and weighed. It is then redissolved in nitric or muriatic acid, and the iron, manganese, alumina, and phosphoric acid separated and determined as in I. §§ 14, 18, 22, 23.

The filtrates and washings being mixed, determine in them lime, magnesia, potash, and soda (I. §§ 9 and 10).

Lastly, determine in a fresh portion of the sample the amount of carbonic acid (I. § 8, *b*).

Marl is analyzed in the same manner. The comparative value of limestones depends upon the purpose to which they are to be applied. For agricultural purposes, their value is improved by the presence of phosphoric acid, potash, and magnesia. (The vulgar prejudice against the latter ingredient arises simply

from the fact that burnt magnesia retains its causticity longer than burnt lime. It should be remembered that wheat takes up more magnesia than lime.) For manufacturing purposes, the nearer it approaches a pure carbonate of lime, and the whiter it is, the better. Lime for hydraulic cements requires a considerable proportion of alumina, some samples containing 20 per cent.

§ 143. GYPSUM; ALABASTER.

Dry a weighed portion, finely powdered, over sulphuric acid. The loss shows hygroscopic moisture. Ignite the residue in a platinum crucible, and note the loss as combined water. Dissolve in muriatic acid with much water; filter off, wash, dry, ignite, and weigh the insoluble residue of sand and clay. Heat the filtrate with a little nitric acid, or treat it with chlorine gas, and add ammonia, as in II. § 142. The precipitate (sesquioxide of iron) is filtered off, washed, dried, &c., and weighed. The washings are added to the filtrate, which is then divided into two portions. In one of these determine lime, in the other sulphuric acid.

The presence of iron renders gypsum of little value for plaster, and entirely unfits it for its various uses amongst millers, bakers, confectioners, and manufacturers of painters' colours.

§ 144. CARBONATE OF BARYTES, NATIVE.

Dry at a gentle heat, and ascertain the moisture lost. Dissolve a weighed portion in dilute hydro-

chloric acid. Sand, clay, and sulphate of baryta may remain undissolved. These impurities are filtered off, dried, &c., and weighed. In the filtrate, baryta is determined by adding sulphuric acid, as in I. § 11, keeping the solution very dilute. From the filtrate previously heated with a little nitric acid, or treated with a current of chlorine gas, oxide of iron, if present, is thrown down by adding an excess of pure ammonia, and filtering with as little access of air as possible.

§ 145. CLAYS.

Clays for technical purposes should contain as little as possible of any substance but silicate of alumina. Treat a portion with dilute muriatic acid, digest, filter, and dry the residue. The smaller the loss, and the less effervescence caused on addition of the acid, the better is the clay for fire-bricks, earthenware, &c. See also I. § 23.

§ 146. CHROME ORES.

Powder finely and mix with three or four parts of quicklime previously slaked with caustic soda, dried, and calcined. Add about one-quarter part nitrate of soda, and ignite for two hours, stirring every quarter of an hour with a platinum wire. The mass, when cold, is extracted with water; and to the insoluble portion sulphuric acid is added, diluted with twice its bulk of water. When the whole is removed from the crucible, a little alcohol is added to the solution, which renders the bulk of the sulphate of lime nearly insoluble. The whole is thrown on a filter, and washed

with very weak alcohol, which dissolves the bichromate, and leaves on the filter sulphate of lime, and any unattacked portions of ore. The latter, freed from gypsum by washing with plenty of boiling water, is recalcined as above. To the filtrate is added a mixture of ammonia and oxalate of ammonia. Filter again, wash thoroughly, add the washings to the clear liquid, and concentrate. The amount of chrome may now be determined by acidulating with hydrochloric acid and boiling with alcohol. The application of heat is continued until the alcohol is driven off. Ammonia is now added as long as a precipitate is formed, and the solution heated until it becomes colourless. Filter, &c., and ignite carefully in a well-covered platinum crucible, and weigh as sesquioxide of chrome.—*Calvert.* Or—

Powder very finely in an agate mortar, and mix with ten to twelve times its weight of fused bisulphate of potash. Fuse the mixture in a platinum crucible, and keep it at a gentle red heat for about half an hour. Place the crucible, when cold, in water to extract the saline mass. Boil the whole with an excess of carbonate of potash. Filter the precipitate, wash, dry, and fuse it with a mixture of equal parts of nitre and carbonate of soda for about fifteen minutes in a silver crucible. Dissolve, when cold, in water, filter, and in the filtrate determine the chrome as above by boiling with hydrochloric acid and alcohol, &c.—*Hunt.*

To detect the presence of chrome in minerals, furnace-products, &c., a portion of the substance in

very fine powder is treated as in either of the methods directed above for the quantitative assay of chrome ores. The aqueous extract in which chrome, if present, is contained as chromate of alkali, will have a yellowish colour, and give yellow precipitates with chloride of barium and nitrate of lead, both soluble in nitric acid, and both, if strongly heated with charcoal, or if boiled in hydrochloric acid, turning green. Precipitates supposed to contain chrome may be ignited with a mixture of carbonate of soda and nitrate of potash, and the residue extracted with water, and tested for chromic acid as above.

Liquids, and bodies capable of being dissolved—either in acids or alkalies—in the moist way, and supposed to contain salts of chromium, may be tested by various procedures.

a. The substance is treated with a solution of hypochlorous acid.

b. The substance is dissolved or suspended in a dilute solution of caustic alkali.

c. The substance is treated with a solution of permanganate of potash in an acid, neutral, or alkaline solution.

d. The substance is heated, along with peroxide of lead (or peroxide of manganese), either in a solution of caustic alkali or in acidulated solutions.

By all these methods sesquioxide of chrome or any of its salts may be converted into chromic acid. Free hydrochloric acid delays or even hinders the conversion of chrome into chromic acid.

The most delicate test for chromic acid is as follows :

—Prepare some crude binoxide of hydrogen (oxygenated water) by rubbing up some binoxide of barium (or calcium) in a porcelain mortar. The thin paste obtained is poured by degrees into some hydrochloric acid diluted with about three times its weight of water, keeping constantly stirring. The liquid thus obtained will keep for some time. To apply the test, about 125 grain measures of the binoxide of hydrogen solution are poured into a narrow test-tube, and covered with a layer of ether about 2-10ths of an inch in depth. The solution suspected to contain chromic acid is now poured, little by little, into the test-tube. The tube, after each addition, is closed with the thumb, and gently inverted, without shaking. If chrome was present, perchromic acid will be formed, and dissolve in the ether with a blue colour, which is not permanent.—*Barreswill and Storer.*

§ 147. VANADIUM IN SLAGS AND FURNACE-PRODUCTS.

Powder the substance very finely, mix with one-fourth of its weight of nitre, and heat it to a cherry-red for some hours in Griffin's gas-blast furnace. Digest the coherent mass in boiling water, and filter. Carefully neutralize the filtrate with pure nitric acid, and precipitate with acetate of lead, adding sulphurous and dilute sulphuric acids until the resulting precipitate is white. Filter and boil with caustic potash, which precipitate the chrome; filter and boil with hydrosulphate of ammonia, or precipitate in the cold with SH; collect the precipitate of sulphuret of vanadium, and fuse at a low red-heat with twice its

weight of nitre. Dissolve in water, filter, and immerse pieces of sal-ammoniac in the solution for three or four days. The crystals of vanadate of ammonia separate in a granular form, and may be converted into vanadic acid by a low red-heat.—*Deck.*

§ 148. IRON.

1. Dissolve in muriatic acid, add very cautiously a few drops of a very dilute solution of protochloride of tin until the solution no longer shows a yellow tint. If nitric or nitro-muriatic acid have been used as a solvent, a larger amount of tin solution is needed to bring all the iron to a state of protoxide. Pour in carefully a standard solution of bichromate of potash from a pourette, stirring well, and bringing from time to time a drop of the solution in contact with a drop of highly diluted red prussiate of potash. As long as any protochloride of iron remains unchanged, a blue colour will be produced; as the action approaches completion, a greenish shade will be communicated, closely followed by a reddish tint, which indicates that the operation is finished. 88.75 grains of bichromate of potash represent 100 grains of metallic iron. If therefore 44.4 grains of bichromate are dissolved in 1000 grain measures of water (the contents of an ordinary pourette), one degree, or 10 grain measures, will represent half a grain of iron.—*Penny.* Or—

2. Mix the dissolved sample with a weighed excess of bichromate of potash, acidulate with sulphuric acid, and heat to boiling. Neutralize with ammonia, add oxalate of soda, place in the inner tube of the

apparatus I. § 8, *b*, and proceed as there directed. If chlorine is present, oxide of mercury must be added. As one equivalent of bichromate of potash evolves six equivalents of carbonic acid, the amount of carbonic acid which should have been evolved, if no reduction had taken place, can be easily calculated; and by deducting from this the quantity actually obtained, the amount of chromic acid employed in oxidizing the iron is arrived at, and from this the iron is deduced. 168·2 parts of iron require, for their conversion from protoxide to peroxide, 148·7 parts bichromate of potash, and consequently diminish the production of carbonic acid by 132 parts. If the iron is present as peroxide, it is first reduced to protoxide by treating the solution with zinc or sulphurous acid. If it occurs in both states, the protoxide is first determined; then in another portion the whole amount of iron, after reduction with zinc or sulphurous acid, and the amount of peroxide of iron is calculated from the difference. — *Vohl*.

§ 149. IRON ORES.

a. Hematites (ores containing sesquioxide only)—From 15 to 30 grains of the ore, finely powdered, are placed in a small flask, with half an ounce of the strongest hydrochloric acid, and heat applied. A solution of protochloride of tin, specific gravity 1·6, is now gradually dropped in until the oxide of iron is entirely dissolved, and the liquid assumes a pale yellow colour. A mixture of 5 parts of the tin solution with 95 parts of water is now added from a

pourette, until the liquid becomes quite colourless. The solution containing the reduced oxide of iron is now largely diluted, and the iron determined as in II. § 148.

b. Magnetic iron ore, roasted clay band or black band, and mixtures of proto- and sesquioxide—

Dissolve in muriatic acid, and estimate the amount of protoxide by bichrome, as above. Then in a fresh portion estimate total iron by the process just described. The former amount, deducted from the latter, leaves the quantity present as sesquioxide.

If it is desired to determine all the constituents of an iron ore, proceed as in I. § 23.

Alumina may be rapidly separated from iron, and determined as follows:—Precipitate the two oxides with ammonia, filter, wash, dry, ignite, and weigh in the usual manner. Powder well in a small mortar, ignite to expel moisture which may have been absorbed, and weigh once more. Determine the iron as directed for hematites, and calculate loss in pulverizing. The difference between the original weight of the precipitate and that of the oxide of iron gives the alumina.

Iron Slags.—Powder very finely, and digest with the strongest muriatic and a little nitric acid, stirring well, and continuing the heat until the whole appears a uniform yellowish mass. Evaporate to dryness, redissolve in dilute muriatic acid, filter off the silica, wash, dry, and weigh, &c.

In the filtrate, determine iron, manganese (if present), lime, magnesia, and alkalies (I. § 23).

§ 150. PHOSPHORUS IN IRON ORES AND METALLIC IRON.

a. Dissolve in nitric acid, add hydrochloric acid, and evaporate to a sirupy consistence. The residue is diluted with water, and the solution poured, drop by drop, into a mixture of protosulphuret of potassium and silicate of potash. To 10 grains of iron should be employed 50 grains sulphuret of potassium, and 10 grains of a soluble glass prepared by fusing 1 part of powdered quartz with $2\frac{1}{2}$ parts of a mixture of 5 parts carbonate of potash and 4 parts carbonate of soda. The precipitate which is formed contains iron as sulphuret, and silicates of lime, magnesia, and alumina. To prevent any sulphuret from dissolving, an excess of recently-prepared carbonate of lead is added before filtration. The precipitate is thrown on a filter, and washed with water containing 1-10th of its bulk of saturated solution of carbonate of ammonia and a little caustic ammonia. The filtered liquid is evaporated almost to dryness, and the residue moistened with hydrochloric acid to separate the silica. The solution contains all the phosphorus as phosphoric acid. Mix with tartaric acid, and determine the phosphoric acid by acetate of magnesia.

b. Dissolve 120 grains of tartaric acid in a little water. Dissolve in another portion of water 210 grains of cyanide of potassium, so as to make a very strong solution. Meantime dissolve 15 grains of the sample of iron in nitric acid, to which a little hydrochloric has been added. The tartaric acid is then poured into the iron, some sulphate of magnesia is

added, then a considerable excess of ammonia, and lastly the cyanide of potassium. A deep red liquid is thus obtained, and sulphuret of potassium is added, drop by drop, until the liquor which first becomes of a light yellow again acquires a deep tint. After some hours a precipitate of ammonio-phosphate of magnesia is deposited along with a little sulphur. It is washed with ammonia water, dried, heated to expel sulphur, and fused in a platinum crucible with a mixture of carbonate of potash and carbonate of soda. The residue is dissolved in water, mixed with tartaric acid and ammonia, and the phosphoric acid thrown down by sulphate of magnesia.

If lime be present, a modification of the process becomes needful. After having commenced the operation as above, instead of sulphate of magnesia chloride of calcium is added, and then sulphuric acid and alcohol. The sulphate of lime is filtered off, and the operation continued as before.

§ 151. CAST-IRON.

a. Determination of Total Carbon.—15 grains of metal clippings are placed in a small beaker, with an equivalent of iodine, say, 72 grains to 15 grains of metal; a little water is added, and the glass, with its cover on, set aside at a common temperature; five to six hours suffice for solution, and only enough water to cover the mixture should be added. Perfect solution is recognized in the dark-brown liquid by the flocculent character of the insoluble matter, as compared with the heavier and metallic particles. If any

of the latter remain unacted on, they will subside heavily to the bottom. In such case, a few more grains of iodine must be added, if the first portion has quite dissolved, and the whole must be filtered as soon as the solution of the metal is accomplished; for a prolonged action of the iodine, as well as too great dilution, may cause the loss of some carbon. Great care must be taken to moderate the reaction, and *slight* additions of water become, therefore, occasionally needful. When it is evident that no fine particles of metal remain at the bottom, the solution is perfect, and the insoluble matter, carbon slag, silica, and some oxide of iron float in the liquid which contains the remaining constituents as iodides. The solution is immediately diluted with water, thrown on a balanced filter, washed with hot water, then with hydrochloric acid to remove traces of sesquioxide of iron, and finally with hot water. The filter is then first dried between layers of blotting paper, and then under the air-pump over sulphuric acid, along with the counterpoise of the filter. The two are then weighed against each other, when the difference of weight shows the amount of insoluble residue. But as this does not entirely consist of carbon, the filter is placed in a platinum crucible, and ignited until every trace of carbon is burnt off, and the residue again weighed. The loss by ignition shows *total carbon* contained in the iron.

b. Graphite or Allotropic Carbon.—15 grains of the sample are digested at a gentle heat in hydrochloric acid of specific gravity 1.1. In a few hours the soluble ingredients of the sample will have dissolved,

leaving the carbon in flakes or lumps along with some silica suspended in the liquor. The sediment is to be collected upon a balanced filter, washed with hot solution of potash, of specific gravity 1.29, to remove humus and extractive matter, then with dilute muriatic acid, and finally with hot water. The last washing must be perfect. The filter is then dried; first between layers of blotting paper, and then over sulphuric acid under the air-pump. It is then weighed. The filter and contents are now put in a platinum crucible, cautiously ignited over a powerful gas lamp, and again weighed. The weight of the residue is then deducted from the original weight of the contents of the filter. The difference shows the amount of graphite.

Great care must be taken to burn off every trace of the carbon, and all draft must be avoided.

c. Combined Carbon.—Its amount is found by deducting the weight of graphite from that of the total carbon.

d. Silica and Slag.—15 grains of clippings, accurately weighed, are put in a wide-mouthed flask. By means of a well-fitting cork, pierced with two holes, a tube-funnel and a drying-tube filled with chloride of calcium are adapted to the flask. A glass tube, drawn out at one end to a fine point, is adapted to the outer end of the drying-tube. Perfectly pure hydrochloric acid, of specific gravity 1.06, is poured into the flask through the funnel-tube, and the flask slightly warmed to start the action; gases are soon generated, and after having passed over a sufficiently long time to expel all common air from the flask and tubes, a light

is applied to the orifice of the exit tube, so as to ignite the jet of gas. A piece of cold white porcelain is held against the flame. If after five minutes' combustion no adhesive spot is formed upon the porcelain, there is no arsenic present. If a large distinct spot appears promptly, it must be quantitatively determined along with the sulphur (see below). In either case the apparatus is taken to pieces, the flask covered with a watch-glass, placed on the edge of the sand-bath, and allowed to stand there until the solution is complete. The liquid is now evaporated to dryness, re-digested in very dilute hydrochloric acid, filtered, washed with hot water and hydrochloric acid, and lastly, with hot water. It is then dried, the residue ignited in a platinum crucible until all carbon is burnt off, and the residue weighed as *silica* and *slag*. The weighed mass is put in a small silver capsule and boiled for two or three minutes with half a fluid ounce of caustic potash liquor at 1.10 specific gravity. It is allowed to settle, and then filtered, and washed with hot water until a drop of the liquid which passes through leaves no residue when evaporated to dryness on a piece of platinum foil. The filter is dried, ignited, and weighed. This gives the weight of *slag*, which, deducted from the joint weight of slag and silica as above obtained, leaves the weight of *silica*, from which its equivalent of silicum may be calculated.

e. Calcium, Magnesium, Sodium, Potassium, Cobalt, Nickel, Iron, Manganese, Aluminium, Copper, and Phosphorus.—Place 75 grains of clippings of the sample in an 8-ounce beaker, drench them with fuming

nitric acid, cover, and set aside in a cool place. After twelve hours more nitric acid is added, and the beaker placed where it may not be heated higher than 212° Fahr. When the solution is perfect, as may be known by the flocky appearance of the insoluble matter, the cover is removed, and the liquid gradually evaporated to dryness. Hydrochloric acid is now added, the cover replaced, and digestion continued until solution is again effected. Phosphoric acid, iron, chrome, manganese, aluminium, calcium, magnesium, sodium, potassium, cobalt, nickel, and copper (if present), remain in solution. The liquid is now diluted with water, filtered, and the residue well washed with hot water. The filtrate we call A, the residue α . The latter is reserved to be examined for tin and titanium.

To A, add ammonia free from carbonate in excess. Alumina, chrome, manganese, iron, and phosphoric acid are thrown down. Filter immediately; wash well with hot water. The residue, β , is set aside.

The filtrate, B, is concentrated by evaporation, mixed with twice its bulk of absolute alcohol, and a few drops of sulphuric acid added to throw down the lime. The precipitate of sulphate of lime is filtered off, washed with dilute alcohol, ignited, and weighed, and the amount of *calcium* calculated from its weight.

The filtrate from this operation is evaporated very cautiously to dryness, and heated for the perfect expulsion of ammoniacal salts. If the sulphuric acid be in excess, a little carbonate of ammonia should be added before heating, but after evaporation. The heating is continued until the weight of the crucible

and contents become constant. This gives the weight of the sulphates of potash, soda, and magnesia, with traces of copper, cobalt, and nickel, if present.

This mass is dissolved in water, and the solution divided into two equal portions. In one portion of the solution sulphuric acid is determined in the usual manner; the result, doubled, representing the amount of that acid in the above-mentioned sulphates. The excess of baryta is now removed from the solution by sulphuric acid, and a current of SH is passed through the liquid, whereby *copper*, if present, is thrown down as sulphuret. The mixed precipitate is filtered off, and the copper determined, if in sufficient quantity, by taking up the sulphuret in a little aqua regia, and proceeding in the usual manner (I. § 74).

The liquid filtered from the sulphuret of copper is tested for *cobalt* and *nickel* by adding ammonia and a few drops of hydrosulphate of ammonia. If a black sulphuret is thrown down, one or both of these metals is present. The black precipitate thus obtained may be re-dissolved and submitted to further tests. These two metals rarely, if ever, occur in ponderable quantities. In the second part of the solution, magnesia and the alkalies are determined as in I. § 9.

The residue, β , containing oxides of manganese, chrome, iron, and alumina, along with phosphoric acid, is carefully transferred to an 8-ounce beaker; the filter being washed with hot water, hydrochloric acid, and again with hot water, to remove every trace from the pores of the paper. The residue is dissolved in

hydrochloric acid and the extract of the filter paper added. The liquid is neutralized with ammonia, and hydrosulphate of ammonia is then added in slight excess, followed immediately by two fluid drachms of caustic potash, of specific gravity 1.30. The iron and manganese are thrown down as sulphurets, whilst alumina and chrome are retained in solution. When the precipitate has quite subsided, the clear liquid is filtered off, and six fluid drachms of caustic potash solution, specific gravity 1.30, are added to the sediment, and the whole digested for a few minutes to remove every trace of alumina and phosphoric acid. It is then diluted with water, filtered on the same filter as employed for the former liquid, and washed with dilute hydrosulphate of ammonium. Call the filtrate G, and the contents of the filter γ .

Manganese.—The filter, γ , contains sulphurets of iron and manganese. The contents are digested in nitric acid, filtered and washed to remove sulphur. From the clear liquid, the iron and manganese are thrown down by ammonia, the precipitate filtered, washed in hot water, and redissolved in muriatic acid. When cold, a slight excess of pure carbonate of baryta is added by degrees, and the whole set aside at common temperatures for twelve hours, being now and then stirred. The iron is now found thrown down, while the manganese remains in solution. The whole is filtered, and washed with hot water. The filtrate is D, and the precipitate δ . If D is at all turbid, it must be evaporated to one-fourth its bulk, and the sediment added to δ by passing the whole

again though the same filter as before. To the clear liquid some sulphuric acid is added, to remove the excess of baryta; the whole filtered with the usual precautions, and washed with hot water. The filtrate is further concentrated by evaporation under a cover of porous paper, tested with a drop of sulphuric acid in proof of the absence of baryta, and mixed with an excess of solution of carbonate of soda. The liquid is well warmed on the sand-bath, the precipitate of manganese filtered off, washed with hot water, dried, and ignited, until the weight of the residue, Mn^3O^4 , becomes constant. From this the weight of the metallic manganese is calculated.

Iron.—The contents of filter δ are transferred with a platinum spatula into a beaker, into which the filter-paper and the spatula are carefully washed with hydrochloric acid and hot water. It is digested with a little more muriatic acid until completely dissolved, the baryta is thrown down with sulphuric acid, and the precipitate of sulphate of baryta filtered off. The precipitate on the filter is well washed, first with muriatic acid and then with hot water, and the washings added to the filtrate. To the clear filtrate, thus freed from baryta, is added ammonia in slight excess; the precipitate of oxide of iron is filtered off, washed thoroughly with hot water, dried perfectly, and the precipitate and the paper ignited separately in a platinum crucible to avoid reduction. The metallic iron is then calculated from the weight of sesquioxide, Fe^2O^3 , obtained.

The filtrate, Γ , contains the bulk of the phosphoric

acid combined with potash. It is digested with nitric acid for some hours, *below* 212° Fahr., to expel ammoniacal salts and promote the settling of sulphur, which after twelve hours' repose is filtered off. After thus removing the sulphur, the filtrate is treated with ammonia in slight excess, the beaker is covered with porous paper, and heated upon the sand-bath. After a time alumina subsides, and is filtered off, washed with hot water, and set aside as ϵ ; the filtrate is E.

Phosphorus.—Phosphoric acid is determined in E by the usual methods, and its corresponding amount of phosphorus found by calculation.

To separate the remaining traces of phosphoric acid from the aluminous precipitate ϵ , the latter is dissolved in muriatic acid, and tartaric acid added until it no longer gives any precipitate with ammonia. Sulphate of magnesia along with muriate of ammonia is then added in solution. The precipitate of ammonio-phosphate of magnesia is allowed to stand for some hours, washed with very dilute ammonia, dried, ignited, weighed, and the amount of phosphorus, calculated from its weight, is added to that from filtrate E.

Aluminium.—Alumina is determined by evaporating the filtrate to dryness, igniting to carbonize organic matter, fusing the residue with bisulphate of potash, taking up in muriatic acid, precipitating by carbonate of ammonia, washing with water, igniting, and weighing. The alumina thus obtained is tested for *chrome*.

Tin and Titanium.—The residue a contains these two metals, if present. If *tin* existed in the metal, it was converted into insoluble peroxide by nitric acid

and evaporation. Its presence is proved by fusing the suspected insoluble residue with carbonate of soda on charcoal, grinding that part of the coal in an agate mortar with water, and washing away all the charcoal and other light matter, when tin will betray itself by silvery streaks produced by the pestle.

The presence of *titanium* is best shown by fusing the whole residue with carbonate of soda, extracting with luke-warm water, filtering, partially neutralizing, and boiling; titanitic acid will be thrown down. A yellowish hue in the soda solution also betrays the presence of chrome. If tin and titanium are largely present they are determined in the usual manner.

Arsenic, Antimony, and Sulphur.—To estimate these bodies, if present, take seventy-five grains of fresh clippings, place them in a 16-ounce beaker, and add a measured ounce of nitric acid. The glass is covered with an inverted capsule, and very gradually heated to boiling. Meanwhile 195 to 225 grains of chlorate of potash in fine powder are gradually added, and the heat is kept up till the insoluble matter begins to float. The clear liquid (containing everything except silica and slag) is evaporated to dryness, redigested in muriatic acid diluted with water, filtered, and the sediment washed with hot water. In the filtrate, which is heated, are placed strips of bright metallic copper, and digested until a fresh surface is no longer tarnished. The slips are then removed, rinsed in pure water, and, when dried, are carefully laid upon a sheet of glazed paper, and the tarnished surface is scraped off with a knife. These scrapings

are then dissolved in aqua-regia, and the arsenic acid estimated as in II. § 43.

The presence of *antimony* may be ascertained by testing the spots deposited on porcelain (see *d*) by a solution of iodate of potash or of chloride of soda, both of which rapidly dissolve arsenical spots, leaving those of antimony untouched.

Sulphur.—After arsenic, if present, has been separated, solution of chloride of barium is poured into the residual liquid, which is then left to stand for a few days. The precipitate of sulphate of baryta is filtered off, washed with dilute muriatic acid and hot water, ignited, and weighed, and from its weight the amount of sulphur is calculated.—*Morfitt and Booth*.

Steel may be analyzed in the same manner as cast-iron.

§ 152. COPPER (*in Ores as Alloys*).

1. Prepare as follows a standard solution of pure cyanide of potassium:—Dissolve two grains of pure metallic copper in nitric acid, evaporate off excess of acid, dilute with water, and add a large excess of ammonia. Then setting the beaker on a sheet of white paper, drop in the cyanide solution with a pourette, stirring frequently. When the deep blue of the ammoniacal solution changes to a lilac-purple the test-liquor must be added very carefully, pausing at intervals, until the colour be very nearly destroyed.

A faint lilac tinge remaining in the liquid will often disappear on standing for some ten minutes exposed to the light. If colour is still found to remain, add

another drop or two, stir well, and let stand once more. Note the number of degrees required to discharge the colour of the copper. If 30 to 40 degrees of the pourette are consumed by two grains of copper, the liquid is of convenient strength.

Having thus ascertained the value of the cyanide solution, dissolve the substance to be tested in nitric acid, expel excess of acid by heat, and add ammonia in large excess. Filter off any metallic oxides which may be precipitated by the ammonia. The precipitate, if bulky, and especially if oxide of iron be present, is very well washed with ammonia, and the washings added to the filtrate. As, however, the oxide of iron can retain much ammoniacal copper in its pores, which it is scarcely possible to expel by washing, it is safer to add the cyanide solution at once to the solution of the ore, without filtration. This method requires a little more time for the oxide of iron to settle after stirring, but is safer.

Others, after having washed the precipitate well, redissolve it in a little nitric acid, dilute with water, supersaturate with ammonia, and filter again, adding the two filtrates together.

In any case the cyanide solution is dropped into the ammoniacal copper liquid, as above, until the colour is very nearly discharged, when the quantity of copper in the substance is easily calculated from the amount of cyanide solution consumed.

This process is applicable for alloys of copper with tin, arsenic, antimony, and lead; for the native oxides, carbonates, sulphurets, chlorides, phosphates,

arseniurets and arseniates of copper, also for the double sulphurets of copper and iron. If silver is present, it must be removed by adding hydrochloric acid to the acid solution of the ore, filtering, adding the washings to the filtrate, and heating again with more nitric acid to expel the residue of hydrochloric. The rest of the process is as above.

Chrome, nickel, cobalt, zinc, and manganese, likewise interfere seriously; as also mercury and arseniate of iron, the latter of which masks the colour. For the separation of these bodies see I. §§ 74 and 76; or—

2. Dissolve in nitro-hydrochloric acid, expel the nitric acid by digestion and boiling with an excess of hydrochloric acid. Drive off as much as possible of this also by evaporation, so that the liquid may be but moderately acid. Put in a clean piece of iron, and allow it to remain until all the copper has been thrown down. Then remove the iron, wash off all particles of copper adhering to its surface, brushing off with a feather any traces which are harder to remove, pour away as much as possible of the ferruginous solution, and wash by decantation with water, at about 110° Fahr., acidulated with 1 or 2 per cent. of hydrochloric acid. When the liquid no longer gives a blue precipitate with red prussiate of potash the washing is at an end, the copper is carefully dried in the same capsule, and weighed. Some, instead of drying and weighing the precipitate of metallic copper, redissolve it in nitric acid, add excess of ammonia, and determine by means of cyanide of potassium. In this case the procedure may be somewhat modified.

The solution of the substance may be more freely acid, and any copper stains adhering to the iron may be dissolved off in nitric acid, and the solution added to the solution of the bulk of copper thrown down.

This method separates copper from the metals which interfere, except silver and mercury. The former, if present, is thrown down by the muriatic acid, and may be filtered off. Mercury will be deposited along with the copper, and may be expelled by strong ignition, after which the copper is taken up in nitric acid, and treated with ammonia in excess, and determined by cyanide of potassium.

The amount of a copper ore taken for analysis may vary from 25 to 10 grains, according to its richness in metal. It should be placed in a small flask and digested in nitric or nitro-muriatic acid. When the first violent action is over, raise the temperature to boiling, and keep it up, adding more acid as required, until the ore (which must have been very finely powdered) appears perfectly dissolved, and the residue of silica takes a white colour. The flask in which the solution is performed should be placed in a slanting position, to prevent loss by spiriting, and should be most carefully rinsed when the contents are poured out.

Solutions of copper, when treated with cyanide of potassium, should not be too concentrated.

Copper slags are carefully pulverized, and a portion (say 100 grains) digested at a boiling heat in nitro-muriatic acid. The solution is supersaturated with ammonia, made up to fill a stoppered bottle of thin white glass. A set of bottles, precisely similar in size

and shape, should previously be prepared as follows:—Dissolve respectively 1-10th, 2-10ths, 3-10ths, &c., up to one grain of metallic copper in nitric acid, supersaturate with ammonia, fill the bottles with these liquids, making up with water, so that the volume of liquid may be exactly equal in all cases. Fix on the stopper of each bottle a label showing its proportion of copper, and arrange them where there is good light. The bottle containing the solution of the slag under examination is placed alongside these, when the amount of copper it contains may be readily determined by comparing the intensity of the tint. If the copper in a slag exceed 1 per cent., determine by cyanide of potassium.—*J. Cameron.*

If all the constituents of a copper slag are to be determined, then, after pulverizing and dissolving as above, evaporate to dryness, take up again in dilute muriatic acid, filter off the insoluble part, wash, &c., and weigh as silica.

The filtrate is meanwhile treated with HS, and the precipitate washed with HS water. It may contain copper, lead, and traces of antimony and arsenic.—See I. §§ 74, 86, and 88.

The clear liquor and washings may contain cobalt, nickel, iron, manganese, chrome, zinc, lime, magnesia, and potash, &c.—See I. §§ 29, 27, and 23.

§ 153. TIN ORES.

1. Weigh out twenty-five grains of the ore, finely powdered, and well dried. Take a Berlin ware crucible, of about half-ounce capacity, put sufficient cyanide

of potassium to fill it about two-thirds full when fused, and heat it to redness by any appropriate gas-furnace. Take off the lid, remove the gas-furnace from under the crucible, and introduce a little of the ore by means of a paper spatula. Replace the lid, and heat three or four minutes; add a little more, and so on until the whole quantity has been introduced. Then heat for ten or twelve minutes longer. Remove the lid. The contents consist of a spongy mass at bottom, with here and there a bubble of gas adhering, while the rest consists of clear fused glassy cyanide. Have ready a dry, clean saucer, into which pour the contents; it forms a cake, which readily separates when cold. The crucible and lid, when cold, are put along with the cake into a small evaporating dish, covered with hot water, and set on the sand-bath to digest. When all soluble matter appears taken up, the crucible is lifted out, and well washed with the washing bottle into the basin, and laid aside on a clean porcelain slab. The lid is treated in the same manner. The basin is set aside to allow all particles of reduced metal to subside. The clear supernatant liquor is then drawn off with a pipette, and the basin is partly filled with distilled water. The liquid is again withdrawn by the pipette. This process is repeated two or three times.

Finally, the crucible and lid are put in the basin, and hydrochloric acid added to dissolve the metals. The crucible is turned with a glass rod, that all adherent particles of metal may be dissolved off. When the action is at an end the liquid is poured upon a

filter, into which the crucible, lid, and basin, are washed with hot water, and filtered into a flask. Lastly, the filter itself is well washed. The contents of the flask are then concentrated by evaporation to about three-fourths of an ounce. Fragments of zinc are then added by degrees till all the tin is reduced to a spongy metallic mass. Hydrochloric acid is then added, and heat applied until the sponge is redissolved. The amount of tin is then determined as in II. § 35. *Hart.* Or—

2. Powder finely, and heat gradually to redness in a current of hydrogen gas ($1\frac{1}{2}$ hours for 15 to 30 grains), cool in the gas, and weigh. Loss = oxygen combined with iron and tin. Dissolve in nitromuriatic acid, and filter off *silica*. Saturate the acid solution with ammonia; add hydrosulphate of ammonia in excess to dissolve sulphuret of tin; digest for twelve hours, filter, wash the precipitate with hydrosulphate of ammonia. The precipitate is boiled in muriatic acid till no smell of HS is perceptible, mixed with nitric acid, again heated, precipitated with ammonia, and dried and weighed as *peroxide of iron*.

From the filtrate the tin is thrown down by acetic acid; the precipitate is washed, dried, roasted at a gentle heat until acid fumes no longer escape, when it is heated to redness. When cold, a little carbonate of ammonia is placed in the crucible, which is heated again, and this is repeated till the weight becomes constant. It is then weighed as peroxide of tin.

Another method of volumetrically estimating tin

is—Dissolve mineral cameleon in three or four times its weight of water, and a sufficient quantity of nitric acid to turn it of a violet red. To find the value of this solution dissolve ten grains of pure tin in hydrochloric acid, and ascertain how many degrees of the cameleon-liquid must be added before a faint rose colour makes its appearance in the solution.

Then, if the substance to be analyzed be a solid, dissolve in an excess of pure hydrochloric acid, to which a few drops of nitric acid have been added. When the tin is dissolved, it is reduced to the state of protochloride by boiling with a little distilled zinc, taking care that there is an excess of hydrochloric acid present. Then the standard solution is dropped in until a pink tint appears, when the amount of tin present in the sample is at once deducted from the number of degrees consumed.

If the body under examination be a solution of a protosalt of tin, the test-liquid is at once added to a weighed quantity.

If a persalt, it is first reduced to a protosalt, by digestion with zinc as above.

If a mixture of proto- and persalts, two operations are required; the amount present as protosalt is first determined, and a second sample is treated with excess of zinc until all tin is precipitated. This precipitate is then washed and redissolved in excess of muriatic acid, and its entire amount of tin is determined in the same way. The first amount, subtracted from the second, shows the amount of tin present as a persalt in the original liquid.

§ 154. URANIUM ORES (*Pitch blende*).

Dissolve in nitric acid, dilute the solution with water, and pass a current of HS through the liquid at a temperature of about 86° Fahr. Arsenic, copper, and lead are precipitated and filtered off. Chlorine water, or hot nitric acid, is added to peroxidize the iron. Tartaric acid is added, and the liquid saturated with ammonia; no turbidity arises. Bicarbonate of soda is now added, and a rapid current of HS is again passed through as long as anything falls down. The sulphurets of zinc, nickel, and cobalt, if present, are filtered off, and washed with a dilute solution of bicarbonate of soda containing HS. The oxide of uranium is found in the filtrate.

Care must be taken to maintain an excess of carbonic acid in the liquid during the passage of the HS. This may be done by adding some fragments of calc-spar or marble to the sulphuret of iron employed to furnish HS.—*Kessler*.

This method is of course applicable to artificial compounds and mixtures containing uranium.

§ 155. ZINC ORES, ALLOYS, AND SALTS.

Prepare a standard solution as follows:—Weigh out and dissolve about three grains of *pure* zinc, place it in a flask, dissolve in muriatic acid, and then add an excess of ammonia mixed with about one-fourth part carbonate of ammonia, and lastly four drops of sesquichloride of iron. Meantime, dissolve some sulphuret of sodium in water, and with it fill a pourette.

Drop this liquid into the zinc solution very carefully until the red precipitate of oxide of iron is seen to blacken. Thus it is found how many degrees of the pourette represent a grain of zinc. The sulphuret of sodium will be found of a convenient strength if 100 grain measures correspond to one grain of zinc.

To determine the quantity of the sulphuret of sodium, put into a flask about as much water as the bulk of liquid in an analysis. Add four drops of sesquichloride of iron—the same strength as that used above—and drop in the solution of sulphuret of sodium until the red precipitate of iron has become black. Note the quantity of solution required, which must be deducted from the amount answering to a grain of zinc, and from the amount used in each analysis.

We have now the following three cases :—

a. Metallic Zinc, pure or along with other metals; also ores, furnace products, and salts, containing oxide, carbonate, sulphate, chloride, &c.—Weigh off fifteen grains, and dissolve in a flask with the aid of a gentle heat in muriatic acid, to which a little nitric has been added. If copper is present, put in a plate of clean iron (II. § 152), and filter. To the filtrate add caustic ammonia mixed with one-fourth carbonate of ammonia in excess, and heat in a shallow porcelain dish. Filter off the precipitate (iron and manganese, if present), wash with dilute ammonia, add to the clear liquid four drops of perchloride of iron as before, and drop in the sulphuret of sodium solution till the red precipitate of iron is blackened. Read off the num-

ber of degrees used, deduct the ascertained amount required for blackening the precipitate of iron, and calculate how much zinc is denoted by the remainder.

b. Silicates and mixtures of Silicates and Carbonates.

—Treat the substance in a porcelain dish with muriatic acid and a little nitric, evaporate to dryness, take up again in dilute muriatic acid, filter off the silica, and proceed as above.

c. Blende (sulphuret).—Reduce to fine powder, and digest in a flask with nitro-muriatic acid until the sulphuret is decomposed. Proceed as above.

If nickel and cobalt are present, as in some blendes, dissolve in caustic potash in place of ammonia.—*Schaffner.*

§ 156. LEAD.

If the substance containing lead be soluble in water or acids, it is dissolved therein, and then mixed with an excess of potash. Galena is thoroughly oxidized with nitric acid, and also supersaturated with potash. Sulphate of lead is finely powdered, and then treated in the same way. In all these cases a solution of hypochlorite of lime is then added in excess, and the whole heated for a considerable time almost to boiling, when the lead is converted into brown peroxide. The precipitate is thrown upon a filter, washed with boiling water, the filter broken with a glass rod, and the precipitate washed into the beaker in which its conversion into peroxide was effected. A standard solution of protochloride of tin is then dropped upon the filter, noting the quantity used, until all portions of

the precipitate still adhering to the filter are dissolved off. The filter is again washed with hot water, and to the mixed precipitate and washings a further known quantity of the same tin solution is added in excess. Some muriatic acid is added, and heat applied until the whole is dissolved. Add a little iodide of potassium and starch paste, and drop in a standard solution of bichromate of potash to determine the excess of tin. The completion of the process is shown by a blue tint appearing. If the relative value of the bichromate and of the tin solution are known (II. § 35), the amount of lead present is easily calculated, since each equivalent of tin consumed (peroxidized) represents one equivalent of lead.

Lead Ores (Galena).

Oxidize the ore, very finely powdered, by means of fuming nitric acid in a large porcelain crucible, covered with a watch-glass, the convex side undermost; after the violent action has subsided, it is placed upon the sand-bath to complete the oxidation. The cover is washed into the crucible, and the mass evaporated to dryness. Pure nitre, amounting to three or four times the weight of the ore, is then added, and the whole heated gradually to fusion and incipient redness, at which temperature it is kept for a few minutes. If arsenic is present, a little carbonate of soda is added after the evolution of nitrous vapours has ceased, but before the heat is raised to redness.

The mass when cool is dissolved in warm water, to which the rinsings of the crucible are added.

Filter and wash. The filtrate contains the sulphur and arsenic in the form of sulphuric and arsenic acids. The former is determined in the usual way (I. §§ 2 and 3, or II. § 6); for the latter see I. § 88, and II. § 43.

The insoluble matter is treated with dilute nitric acid, which extracts the oxides of lead, silver, zinc, and iron present. For the separation of these see the appropriate paragraphs in Part I.

The part insoluble in dilute nitric acid may contain silica, sulphate of baryta, antimoniac acid, and sometimes gold. The latter, with a portion of the antimoniac acid, is taken up by digestion in nitro-muriatic acid, the solution evaporated to dryness, and heated with pulverized sugar-candy till all the antimoniac acid is volatilized.

If the ore contains earthy carbonates, or carbonate of lead, these should be removed by treatment with acetic, or very dilute hydrochloric acid, and the loss of weight noted before applying the nitric acid. The lead, if any, in this acetic solution is determined.

If fluor spar is present, it will be partially decomposed by fusion with carbonate of soda, fluoride of soda being formed, which will accompany the sulphuric and arsenic acids. A part may still be found at last along with the silica, sulphate of baryta, &c., from which it should be removed by digestion in muriatic acid, before extracting the gold by aqua-regia.

If soluble silica is suspected, the nitric acid solution is several times evaporated to dryness to render it insoluble. When antimony is present, the water for

washing should be gradually replaced by alcohol;
or—

Reduce the sample to very fine powder (I. § 25), digest for some hours in a solution of potash at about 130° Fahr., pass in a current of chlorine until all the lead is peroxidized, filter, and wash. In the filtrate the sulphur is determined as sulphuric acid. The precipitate contains peroxides of lead and iron, silver, sulphate of baryta, and silica. Treat with strong hydrochloric acid, sulphate of baryta, silica, and chloride of silver remain insoluble. The latter is extracted by ammonia (II. § 162). The lead and iron in the muriatic acid solution are separated and determined as usual.

§ 157. COBALT AND NICKEL, WITH THEIR ORES.

Proceed exactly as for lead (II. § 156). Each equivalent of tin consumed represents two equivalents of either cobalt or nickel in the metallic state. In other words, for every 59 parts of tin consumed 59·2 parts of cobalt or nickel exist in the substance under examination.—See also I. §§ 35 and 43.

The principal ores of cobalt and nickel are—

Cobalt Pyrites, or Koboldine.

Sulphur,.....	38·5
Cobalt,	43·2
Copper,.....	14·4
Iron,.....	3·5
	<hr/>
	99·6

Glance Cobalt.

Sulphur,	20·08
Arsenic,	43·47
Cobalt,	33·10
Iron,	3·23
	<hr/>
	99·88

Smaltine—Tin-white Cobalt—Grey Cobalt Speiss.

Arsenic,	65·75
Cobalt,	28·00
Iron and manganese,	6·25
	<hr/>
	100·00

Another Variety.

Iron,	11·42
Cobalt,	14·26
Nickel,	1·79
Arsenic,	72·53
	<hr/>
	100·00

Black Earthy Cobalt.

Oxide of cobalt,	19·30
Oxide of copper,	4·12
Peroxide of manganese,	54·34
Water,	22·24
	<hr/>
	100·00

Erythrine—Cobalt Bloom.

Arsenic acid,	38·89
Oxide of cobalt,	37·57
Water,	24·04
	<hr/>
	100·50

Glaucodote.

Sulphur,	20.210
Arsenic,	43.200
Cobalt,	24.774
Iron,	11.900
Nickel,	trace
	<hr/>
	100.084

Copper Nickel—Kupfernickel.

Nickel,	43.80
Arsenic,	53.05
Iron,	0.45
Antimony,	0.05
Copper,	0.32
Sulphur,	2.13
	<hr/>
	99.80

Two furnace products, *speiss* and *zaffre*, are also important sources of nickel and cobalt.

The former consists of—

Cobalt,	3.2
Nickel,	49.0
Copper,	1.6
Arsenic,	37.8
Sulphur,	7.8
Sand,	0.6
	<hr/>
	100.0

These constituents vary, however, greatly in proportion in different samples, and iron, antimony, and

bismuth may also be present, the latter sometimes to the extent of 20 per cent.

Zaffre consists of a mixture of cobalt ores freed to a great extent from sulphur and arsenic by roasting. It may contain—

Oxide of cobalt,.....	13.5
Sesquioxide of iron,.....	35.7
Alumina,	3.3
Silica,.. ..	43.3
Arsenic,.....	0.8
Water and loss,	3.4
	<hr/>
	100.0

Nickel, bismuth, copper, and silver may also be present.

Kupfernickel may be examined as follows:—

Reduce the sample to fine powder; weigh out 10 to 20 grains, and put it by degrees into a flask containing boiling nitric acid. Continue to apply heat until complete decomposition; dilute, and filter off the silica.

Add to the filtrate a large excess of caustic potash or soda, warm gently, and pass a stream of chlorine gas into the liquid till the precipitate is quite black. Filter and wash. The precipitate contains iron, nickel, and traces of copper and lead. Redissolve in a little muriatic acid, dilute, and pass a current of H_2S , or heat with hyposulphite of soda to remove the copper and lead.—See I. § 74. Heat the filtrate with nitric acid to peroxide the iron, which is then separated (I. § 37). The cobalt and nickel are separated (I. § 44).

In the filtrate obtained after treatment with chlorine, the sulphur, arsenic, and antimony are found as alkaline sulphates, arseniates, and antimoniates.—See I. §§ 2 and 88 ; or—

Digest in nitro-muriatic acid ; when thoroughly decomposed, determine sulphur (I. § 2) and silica from the insoluble portion filtered off. Add carefully dilute sulphuric acid to remove any excess of baryta. Filter, treat the filtrate with sulphurous acid gas to reduce arsenic to arsenious acid, expel the excess of sulphurous acid by a gentle heat, and pass a current of HS through the liquid as long as anything is thrown down. Let it stand for some time, filter, wash carefully, and allow the filtrate to stand again in case anything further should be deposited. The precipitate contains antimony, arsenic, bismuth, and copper (if present), and lead. For the separation and estimation of these bodies, see the appropriate paragraphs in Part I. The filtrate contains the iron, cobalt, and nickel, which are separated as above.

Ores of cobalt, and cobaltiferous and nickeliferous furnace products may be examined in the same manner.

Another method for the examination of cobalt and nickel ores is as follows :—

As a preliminary examination, roast a little on charcoal to see if arsenic sublimes, and dissolve another portion, of a few grains in weight, in a test-tube, when the depth of the blue or green colour will give a comparative indication of the richness of the ore in cobalt or in nickel.

Powder finely, and weigh out from 20 to 100 grains, according to the richness of the ore. Place it in a small porcelain crucible, and ignite cautiously to expel arsenic. When no more fumes are evolved, transfer the residue into a 4-ounce flask, sweeping in the last particles with a feather, and put a small funnel in the mouth of the flask to prevent loss by spiriting. Pour in some muriatic acid, and add nitric from time to time, until all heavy metallic-looking particles have disappeared from the bottom of the flask. Decant the clear solution into a 10-ounce beaker, into which the funnel and flask are rinsed by means of the washing bottle. If, as sometimes happens, sulphur remains, entangling portions of undissolved ore, such residue is washed into a capsule, dried, ignited, and then redigested with more aqua-regia, and the whole matter is finally added to the first portion in the beaker.

Add to the solution carbonate of lime in excess, iron, alumina, with arsenic and phosphoric acid are thrown down, whilst the *bulk* of the nickel and cobalt remains in solution. Filter, wash the precipitate partially, receiving the filtrate and washings in a 40-ounce beaker. Wash the carbonate of lime precipitate into another beaker, where it is dissolved in dilute muriatic acid. The clear liquid is filtered from the insoluble parts of the ore (silica), a solution of acetate of soda is added in excess, the whole heated to boiling when the iron, alumina, &c., are thrown down, and the rest of the cobalt and nickel remains in solution. Filter, and wash in boiling water, adding a little sulphate of soda during the washing. This filtrate may still

retain traces of iron; a little acetate of soda is therefore again added, the whole boiled thoroughly once more, and if rendered at all turbid, passed through a filter again. It is then nearly neutralized with ammonia, and finally added to the bulk of the cobalt and nickel solution in the 40-ounce beaker. There should be enough of the acetates of soda and ammonia to convert the whole of the cobalt and nickel into acetates. A current of SH is now passed through the liquid; cobalt and nickel, along with certain other metals, are thrown down, whilst manganese, magnesia, lime, alumina, and soluble silica remain in solution. When the precipitation is complete, the whole is thrown upon a filter; any particles clinging to the sides of the beaker are removed by the aid of the washing-bottle; or, if they adhere too firmly to be thus loosened, by means of a glass rod, covered at its end with caoutchouc, which is afterwards well washed into the filter. The whole is then thoroughly washed with boiling water, dried, detached from the filter, and put into a small beaker of one to two ounces capacity, capable of being covered with a watch-glass. The filter itself is ignited, and the thoroughly burned ashes also put in the beaker. Nitric acid is *cautiously* added (without care loss may be occasioned by the violence of the action), a little heat being applied towards the end of the operation until every thing is dissolved. The solution may still, besides nickel and cobalt, contain zinc, copper, bismuth, and the other metals capable of being thrown by SH from an acid solution. The nitric solution is diluted with water, and a current of

SH passed through it. These metals are thrown down as sulphurets, and filtered off. Zinc may still accompany the nickel and cobalt. To detect and remove this, boil the filtrate to expel SH, add ammonia until a precipitate appears, acidify pretty strongly with acetic acid, and pass SH slowly through the liquid. If a milkiness appear, zinc is present, and the gas is to be slowly passed until the precipitate shows signs of darkening. The liquid is then filtered. A trace of cobalt carried down with it may be separated by a repetition of this process.—See also I. §§ 36 and 45. The filtrate containing only nickel, cobalt, and salts of ammonia is treated with some pure sulphuric acid, and evaporated to dryness in a balanced capsule, and finally heated sufficiently to expel the excess of sulphuric acid and ammoniacal salts. The sulphates of cobalt and nickel are now weighed in a covered crucible.

The only remaining step is the separation of the nickel and cobalt. If the latter is in excess, proceed as in I. § 44 *b*, with the following precautions:—If cyanide of potassium, perfectly free from silica, cannot be obtained, add the solution of the nickel and cobalt to hydrocyanic acid, and add subsequently potash, as free from silica as can be procured. Do not add the oxide of mercury until the cobaltid-cyanide is perfectly changed into cobalto-cyanide, which may be known by the liquid retaining a pale yellow tint when it cools. Instead of throwing down the nickel as pale hydrated protoxide by the help of peroxide of mercury, it may be precipitated as black hydrated sesquioxide

by means of chloride of soda, to which some pure carbonate of soda has been added. When the latter method, which is for some reasons preferable, is employed, the solution of the cyanides is raised to boiling, and the mixture of chloride and carbonate added as long as any precipitate is produced. Both the hydrated protoxide and sesquioxide, when dried and ignited, yield protoxide, 37·5 of which represent 29·5 of metallic nickel.

If the nickel in the mixed oxides appears decidedly in excess, add to the perfectly neutral solution of the two a solution of chloride of lime as long as it gives an *immediate* black precipitate, and somewhat beyond this, since a black precipitate forming even after some minutes, often contains a trace of cobalt. This black precipitate contains all the cobalt and some nickel. It is filtered off, washed, redissolved, and the cobalt separated out as above. The liquid, during addition of the chloride of lime, must be from time to time neutralized by adding milk of lime.—*Hadow*.

Oxide of cobalt should yield with pure hydrochloric acid a fine blue solution. If the liquid have a greenish cast, nickel and iron are probably present. The better to compare the colours, a known weight of perfectly pure oxide of cobalt may be dissolved in pure hydrochloric acid, the solution made up to a given bulk, and preserved in a stoppered bottle. An equal weight of any sample under examination is dissolved in the same way, put in a bottle of equal size and similar shape, and made up to the same bulk.

A current of HS should produce no precipitate in

the acid solution. If a black precipitate falls, copper or lead is present; if yellow or orange, arsenic or antimony.—See part I.

If the impurities in commercial oxide of cobalt amount to 2 per cent., it is generally rejected in the market.

There is no very simple or rapid method of detecting cobalt and nickel in minerals or furnace-products. The suspected substance may be digested in aqua-regia, the filtered solution freed from excess of acid by concentration, and moderately diluted with water. A current of SH is then passed through the liquid, as long as anything is precipitated, to remove copper, arsenic, antimony, bismuth, and silver, &c. The precipitate is filtered off, heated to expel free SH, digested with excess of nitric acid to peroxidize any iron present, which has been reduced to protoxide by the SH. Ammonia is then added in excess, the precipitate, if any, produced; sesquioxide of iron is filtered off. The filtrate is now concentrated, the ammoniacal salt destroyed (I. § 10), and the residue heated on the oil bath to about 460° Fahr. The dry residue, when cold, is treated with weak nitric acid, without heat. Manganese, if present, remains undissolved. The solution will contain cobalt and nickel, if both are present. Special tests may now be applied.—See Appendix IX. Alkaline nitrates give a characteristic yellow precipitate with nitrate of cobalt.

If the proportion of these metals be large, they may be detected at once by the rose colour peculiar to the acid solutions of cobalt, and the grass-green of those

of nickel, the latter being further distinguished from copper by the action of ferrocyanide of potassium, which does not, as with the latter, produce a reddish-brown, but an apple-green precipitate, which dissolves in ammonia with a pale red colour.

§ 158. ANTIMONY.

Dissolve in muriatic acid, adding alcohol if needful; add chlorate of potash, and leave in a moderately warm place till the smell of chlorine has wholly disappeared. The temperature of the fluid being a little above 104° Fahr., add a known excess of a solution of protochloride of tin of known strength; stir well, and let it stand for a few minutes. Add a little iodide of potassium and starch-paste, and determine the excess of tin not peroxidized by a standard solution of bichromate of potash, the completion of the process being indicated by a blue tint appearing in the liquid. Every two equivalents (187.64 parts) of protochloride of tin consumed will indicate one equivalent of metallic antimony, or 129 parts. All substances capable of peroxidizing proto-salts of tin must, of course, have been previously removed.—*Streng.*

The principal ore of antimony is the native sulphuret (grey antimony ore). It occurs also along with the sulphurets of lead and copper, and in smaller proportions it is a constituent of many metallic ores.

It may be examined by boiling a well-pulverized portion in aqua-regia, and filtering off the undissolved matter, sulphur, and silica, and probably some sulphate of lead, if lead was present in any great amount,

the sulphur having been oxidized by the aqua-regia. Wash the insoluble matter on the filter to remove all free acid, and then digest it in tartrate of ammonia. Sulphate of lead, if present, will dissolve, whilst silica, free sulphur, and sulphate of baryta remain insoluble. If the tartrate of ammonia holds any sulphate of lead in solution, it may be detected by passing into it a current of SH , which will give a black precipitate.

The original filtrate, containing the soluble matter in the sample, is now neutralized with ammonia, and then mixed with an excess of hydrosulphate of ammonia, containing as much sulphur as it can be made to take up. Sulphurets of lead, copper, and iron remain insoluble, while sulphuret of antimony and arsenic, if present, dissolve, and may be filtered off from the insoluble matter.

To ascertain whether arsenic is present, dilute with water, add acetic acid in excess, and boil. The sulphurets are thrown down along with free sulphur. Filter, mix the precipitate with excess of a mixture of equal parts nitrate of potash and carbonate of soda. A little of the same mixture is melted in a clean iron crucible, and the substance is very gradually introduced, until the whole is in a state of fusion. The melted mass is then extracted with boiling water, filtered, acidulated with pure nitric acid, and tested with ammonio-nitrate of silver. If a red-brown precipitate appears, arsenic is present.

Metallic antimony (regulus) contains a variety of impurities. If *sulphur* is present, the powdered metal, when heated with strong muriatic acid, gives off SH

gas, which may be known by its blackening a piece of paper smeared with moist carbonate of lead. If it contain *potassium* or *sodium*, the powdered sample blues red litmus paper, if moistened. *Arsenic* causes the regulus when fused to emit a garlic odour, and may be detected by the method given above for arsenic in ores of antimony. *Lead*—the powdered metal, boiled nearly to dryness with nitric acid, and mixed with water, yields, on filtering, a liquid which gives a white precipitate with sulphuric acid. If sulphur is also present, then, on treating the lead with hot nitrate acid, the lead remains in the form of sulphate. If the antimony in the residue is dissolved out in warm hydrosulphate of ammonia, black sulphuret of lead remains. *Iron*—the sample in fine powder is heated to redness with thrice its weight of nitre, and washed with boiling water. If the yellow residue is then treated with muriatic acid, iron may be detected in the solution by the usual tests.—See Appendix. *Copper*—when the lead has been thrown down by sulphuric acid, copper, if present, remains in solution, and may be detected by adding ammonia, with which it gives a blue solution.

§ 159. BISMUTH.

If the solution be colourless, a standard solution of bichromate of potash is dropped in, until further addition ceases to precipitate chromate of bismuth, and a yellow tint appears in the liquid. If the solution is coloured, make a number of spots of a strong solution of acetate of lead upon a white porcelain plate,

and, after each addition of bichromate to the solution under examination, insert a pointed glass rod, and bring it in contact with one of the lead spots. As long as any bismuth remains in solution, no change will take place; but as soon as the bichromate begins to be in excess, a yellowish milkiness appears in the spot. The liquid should be kept warm during the operation. 71.35 grains of bichromate of potash represent 100 grains of metallic bismuth. Lead and baryta, if present, are removed by means of sulphuric acid.—*Pearson*.

The principal impurities in metallic bismuth are arsenic, iron, nickel, copper, lead, silver, tin, and sulphur. The chief ores of bismuth are the carbonate (bismuth-ochre); sulphuret (bismuthine); oxide (also called bismuth-ochre); plumbo-cupreous sulphuret (Aikenite); telluret (Bornine). It also occurs native.

Ores, alloys, &c., of bismuth are dissolved in hot nitric acid. After the excess of acid has been expelled by evaporation, the solution is moderately diluted with water. An excess of water, unless very much free acid is present, throws down the greater part of the bismuth as an insoluble sub-salt. By this reaction it is distinguished from all metals except antimony and tin. From these it may be known by its giving a yellow precipitate with alkaline chromates.

§ 160. MERCURY.

Native or metallic mercury may often be recognized by the microscope in minerals where it occurs. Mercurial compounds, if soluble in water or acid (prefer-

ably muriatic), are detected by placing a drop of the liquid upon a clean piece of gold foil, and bringing an iron or zinc point in contact with the gold where the drop lies. A white stain will appear, which may be volatilized off by placing the gold foil in a small test-tube, and applying heat; or—

Place upon a clean, bright copper plate, a portion of the suspected body (which need not be dissolved), and moisten with a drop of a strong solution of iodide of potassium. A white stain will appear on the copper if mercury is present.

Mercury, when mixed with organic matter may be mixed with an excess of moist carbonate of soda, and the mixture heated to near redness in a small test-tube. Mercury sublimes, and on breaking up the tube, may be recognized by a lens amongst the empyreumatic matter.

The substance containing mercury is dissolved in muriatic acid; a known excess of chloride of tin is then added with the pourette; the whole is heated until the mercury is entirely reduced, and has collected in solid masses, which takes place at a temperature below boiling. The clear liquid is decanted off into a beaker, the precipitate washed several times and the washings added, and the excess of protochloride of tin is determined by bichromate of potash. In proto-salts of mercury, every equivalent of metallic tin consumed represents one equivalent of metallic mercury, or in sub-salts two equivalents. If the substance is insoluble in muriatic acid, it is treated with aqua-regia, and the solution afterwards digested with

excess of muriatic acid, until all nitric acid is expelled. If insoluble in all acids, it is mixed with an excess of moistened carbonate of soda in a small retort fitted with a receiver, and heated to dull redness. The contents of both retort and receiver are extracted with water, the former being broken up, the residue treated with hot nitric acid in excess, and the solution thus obtained is digested with excess of muriatic acid.

The mercury of commerce sometimes contains traces of tin, lead, zinc, and arsenic. If dissolved in nitric acid, the solution evaporated to dryness, and the residue ignited, nothing should remain. Any residue insoluble in excess of boiling nitric acid is tin. Zinc and arsenic, if present, may be detected by agitating the sample in cold dilute nitric acid. The impurities, except tin, dissolve, and may be detected in the decanted solution.

Pure mercury should not become covered with a black powder if shaken with air in a bottle; and if allowed to run down a gently sloping surface, it should form globular drops, and not tailed or worm-like masses.

Mercury is found native as sulphuret (cinnabar), and alloyed with silver (native amalgam).

The amount of mercury in an ore or mineral may often be determined with sufficient accuracy by igniting the well-pounded sample in a small iron retort along with excess of quick-lime and iron filings. The beak of the retort is placed so as just to touch the surface of a little water in a beaker, into which the mercury distils.

§ 161. MANGANESE.

To detect manganese in minerals, &c., place a small portion of the substance—in fine powder, if a solid—upon a slip of silver foil, add to it a drop of a strong solution of caustic potash (equal weights of alkali and water), boil over the lamp to dryness and raise the heat. The green manganate of potash appears on the foil, which dissolves in a drop of water with a green colour, and subsequently turns red; or—

Add a little powdered sulphur to an equal bulk of the powdered substance on a slip of platinum foil, heat to redness, and continue the heat for some time. Treat the mass with a few drops of water, and filter. If manganese is present, a white precipitate will be formed on adding a solution of yellow prussiate of potash.

By these tests manganese may be detected in minerals, soils, ashes of coal, peat, vegetables, and of dyed fabrics. The peroxide is powdered, treated with excess of the standard solution of protochloride of tin, and the whole heated with muriatic acid until dissolved. Proceed as with lead (II. § 156). Here, also, each equivalent of tin consumed represents one equivalent of metallic manganese.

Other compounds of manganese are prepared with potash and hypochlorite of lime, exactly as directed for lead.—*Streng.*

See also II. § 7.

§ 162. SILVER ORES; ARGENTIFEROUS GALENAS; COMPOUNDS
AND RESIDUAL PRODUCTS CONTAINING SILVER.

Take from 10 to 300 grains, according to the probable per centage of silver, pulverize finely, and boil in a porcelain dish with nitric acid, diluted with three times its bulk of water. The sulphur which separates is removed, and the liquid mixed with a large excess of ammonia, filtered, and the precipitate washed with ammoniacal water. The filtrate and washings are mixed with an excess of muriatic acid, along with a few drops of nitric acid, the precipitate, which takes some time to form where the quantity of silver is small, is washed by decantation, and is finally dried and ignited in a counterpoised porcelain capsule, and weighed as chloride of silver.—*Mène*.

Many poor silver ores, especially argentiferous copper and iron pyrites, before undergoing this process should be roasted. For this purpose 300 to 500 grains of the pulverized ore are placed in an open crucible, and submitted to a very low heat, constantly stirring. Great care is required, especially at first, to prevent the mass from fusing. The roasting is continued until the emission of sulphurous fumes has almost ceased. The roasted mass is then fused with a known excess of metallic lead (the amount of silver in which must be previously determined), stirring frequently until the melted mass no longer adheres to the iron stirring rod. It is then allowed to cool, the crucible broken up, the button of argentiferous lead separated from the slag, and treated as above.

Ores that do not readily dissolve in nitric acid may be treated with aqua-regia, or nitric acid may be used alone at the outset, and a little hydrochloric acid added afterwards.

Certain ores of silver, such as the native chlorides, bromides, chloro-bromides, and iodides, resist the action of all acids. They may be mixed with double their weight of a mixture of five parts carbonate of potash and four parts carbonate of soda, and the whole then heated in a small porcelain crucible. The silver is reduced long before the alkali is perfectly melted. When the crucible is cool, the alkali is washed out with water, and the insoluble matter treated with nitric acid. The silver dissolves, and is determined as chloride; or—

The ore, in the finest possible state of division, is put in water with some clean zinc or iron, and a drop or two of sulphuric acid. The precipitate of metallic silver is washed by decantation, and redissolved in nitric acid.

Chloride of silver may also be reduced by gently heating in a current of hydrogen gas, and the metallic silver dissolved in nitric acid.

§ 163. GOLD IN MINERALS AND ALLOYS.

The presence of gold in ores or minerals may be qualitatively ascertained as follows:—Digest about five grains of the sample, finely powdered, in strong nitric acid for the purpose of removing the sulphurets. The insoluble residue is well roasted on a slip of platinum foil to drive off any residue of sulphur, and is

next digested in aqua-regia. When the solution thus obtained has become clear, take a drop or two by means of a glass rod, and place it upon a piece of thin white porcelain, so as to form a streak. Warm the porcelain over the flame of the lamp; and when the liquid has evaporated, throw upon the spot a jet of flame by means of the blow-pipe. If any gold be present, a beautiful and characteristic purple stain will be produced, and will almost entirely disappear when the heat is withdrawn. The same colour can be made to appear and disappear at will, by alternately heating and cooling the spot.

To determine the amount of gold in a mineral, take of the substance, in fine powder, a known quantity, varying according to the presumed richness of the sample. Expel sulphur, if present, by careful roasting. Treat the residue with dilute nitric acid as long as any action takes place. Pour off the solution and wash by decantation. Treat the residue with aqua-regia, pour off the solution, wash the residue by decantation, mix the solution and washings, concentrate to a small bulk, add a little oxalic acid, and then pour in gradually mon carbonate of soda enough to convert the gold into aurate of soda. Now add crystallized oxalic acid in very large excess, and boil quickly. All the gold is thrown down as a beautiful yellow, spongy mass. This is boiled several times, first in dilute muriatic acid and then in pure water, and dried and heated to redness.

White tellurium and the telluride of gold and lead are soluble in nitric acid; graphic tellurium in aqua-regia.

Ores and alloys of gold which contain not more than 15 per cent. of silver, are soluble in aqua-regia; but if the silver is in larger proportions, the particles are soon enveloped in a coating of chloride of silver, and the operation is entirely, or almost, arrested. If the gold be under 20 per cent., pure nitric acid will dissolve out the silver, leaving the gold untouched. If the proportions are intermediate, the alloy or ore is fused with three times its weight of pure metallic lead (obtained by reducing the acetate of lead), and treating the alloy thus formed with nitric acid, when silver, lead, &c., are dissolved, leaving the gold untouched.

Gold may also be precipitated from a solution containing excess of muriatic acid, by adding oxalic acid or protochloride of iron, and from a solution containing excess of alkali, by passing sulphurous acid gas through the liquid. See also I. § 79.

§ 164. PLATINUM GRAINS.

A weighed quantity of the ore (about 30 grains) is placed in a small glass retort, to which is fitted a receiver kept cool, treated with nitro-muriatic acid, and distilled until the residue in the retort congeals on cooling. This is dissolved in a very little water, and carefully decanted off. The distillate is poured back upon the undissolved residuum, and treated as before, until the whole be dissolved. The distillate contains osmium. If the metallic solution smells of chlorine, it is allowed to digest until this disappears,

and any turbidity is redissolved. The solution is passed through a weighed filter, which retains undissolved particles of sand and osmium of iridium. The filtrate is mixed with double its bulk of alcohol at specific gravity 0.833, and a strong solution of chloride of potassium added. A precipitate appears, which is filtered, washed with dilute spirit (containing 60 per cent. of alcohol), to which a little chloride of potassium has been added. Wash until the liquor which passes is no longer affected by H₂S. The washed precipitate is dried, and well mixed with an equal weight of carbonate of soda. The filter is burnt separately; its ashes mixed with a little carbonate of soda, and added to the rest. The whole is gently heated in a porcelain crucible till it is black throughout. The residue is washed with water, and afterwards with dilute muriatic acid, and the undissolved matter filtered, washed, dried, and ignited, burning the filter apart. The mass is then weighed, mixed with five or six times its weight of bisulphate of potash, and fused in a platinum crucible with a well-fitting cover. When cool, it is extracted with boiling water, and the residue again treated in this manner five or six times, till the salt ceases to acquire a colour. The undissolved residue is washed, ignited, and weighed; 71 per cent. of the loss is metallic *rhodium*. The solution is mixed with carbonate of soda in excess, the liquid dried, and the residue ignited, and extracted with water; oxide of rhodium remains, and is filtered, washed, ignited, and reduced in a current of hydrogen, and weighed. It is then treated with nitro-muriatic acid, the solution

neutralized, and cyanide of mercury added. If a precipitate is formed (*palladium*), its amount is deducted from that of the rhodium.

The residue, undissolved by the sulphate of soda, is digested first in very dilute nitro-muriatic acid, which is then decanted off, and replaced by concentrated, to which some chloride of sodium has been added. The undissolved matter is washed first with weak common salt, then with weak salammoniac. The residue is burnt with the filter, reduced by a current of hydrogen, and weighed as iridium. The solution is mixed with carbonate of soda, dried, and ignited. The mixture of platinum and peroxide of iridium thus obtained is washed with water, and then treated with nitro-muriatic acid, when peroxide of iridium is left. From the solution ammonia throws down a little more peroxide of iridium. The peroxide of iridium is reduced, and the amount of metal thus obtained added to what was found before. To find the quantity of platinum, the weight of the peroxide of rhodium is subtracted from the weight of the mixed platinum and rhodic and iridic oxides (the mass previous to the action of bisulphate of potash). Add 12 per cent. to the weight of the metallic iridium, and deduct the sum from this same joint weight. The remainder is platinum.

The alcoholic filtrate and washings are poured into a flask, treated with HS, stoppered, and left to stand for twelve hours in a warm place. Filter, evaporate the alcohol from the filtrate, and mix together the precipitates obtained. Whilst the alcohol is evapo-

rating, a fatty deposit is formed which cannot be removed by water. This is taken up by ammonia, after draining off the solution. This solution is evaporated to dryness in a platinum capsule. The precipitates of sulphurets are placed in the same capsule, and the whole is roasted as long as sulphurous acid is given off. Strong hydrochloric acid is added, which dissolves copper and palladium, leaving rhodium, iridium, and a trace of platinum. The hydrochloric solution is mixed with nitric acid and chloride of potassium, and evaporated to dryness. The dry residue is treated with alcohol at 0.833 specific gravity, which extracts the copper as potassio-chloride. The palladium salt is thrown on a balanced filter, washed with alcohol, and dried. It contains 28.84 per cent. of palladium. The copper in the alcoholic solution is determined as usual, after expelling the alcohol by heat.

The sulphurets which escaped solution in hydrochloric acid are treated with bisulphate of potash as above described. When all the rhodium is thus extracted, nitro-muriatic acid will remove a trace of platinum, and iridic oxide remains.

§ 165. PLATINUM ORES (*Deville's Method*).

1. Take a small crucible with smooth sides, and melt in it a little borax, to glaze the inside. Now place in it 110 to 150 grains of pure granulated silver, and 31 grains of the ore, fairly taken and accurately weighed. Over the ore place 150 grains of fused borax, and one or two small pieces of wood-charcoal.

The silver is now fused and kept for some time a little above its melting-point, that the borax may dissolve out all vitreous matters. The crucible is allowed to cool; and, when cold, the button containing the metals is detached, and if needful digested for a short time in hydrofluoric acid to remove traces of borax. It is next heated to dull redness and weighed. This weight, subtracted from the joint weights of silver and ore taken, gives the *sand*.

Two or three portions of each sample should be examined for sand at the same time, to insure greater accuracy.

2. Thirty-one grains of the ore are weighed out very carefully, and digested in aqua-regia at 158° Fahr. till all the platinum is taken up. The aqua-regia is renewed occasionally for twelve to fifteen hours, or till it no longer becomes coloured. The operation is performed in a large beaker with a cover to prevent loss. The solution is very carefully decanted off. The insoluble residue is washed by decantation, dried, and weighed. If the liquid carries with it any small floating particles, these may be collected on a filter, and when dried added to the dried residue in the glass. By subtracting from the weight of this residue the weight of sand, as determined in No. 1, we find the *osmide of iridium*.

3. The aqua-regia solution of No. 2 is evaporated to dryness at a low temperature, and the residue taken up in a little water. If it does not prove entirely soluble in water, more aqua-regia must be added, and the evaporation repeated. To the aqueous solution is

added twice its volume of pure alcohol, and a large excess of crystals of chloride of ammonium. It is now gently warmed to dissolve the crystals, stirred, and set aside for twenty-four hours. An orange or reddish-brown precipitate is formed, containing the bulk of the platinum and iridium, a little remaining in the liquid. The precipitate, *a*, is filtered, washed with alcohol, dried in a platinum crucible, which for security's sake is placed within a larger one, and gradually heated to low redness. The crucibles are now uncovered, and the filter burnt at a low temperature. Once or twice, after burning the filter, a piece of paper soaked in turpentine is placed in the crucible, which is still at a low red heat; by which means the iridic oxide is reduced, and any traces of osmium expelled. The crucible is now kept at a white heat until its weight becomes constant.

The liquid filtered from the precipitate, *a*, is evaporated down until chloride of ammonium crystallizes out in quantity. It is then let cool, decanted, and a small quantity of a deep violet salt is thrown on a filter, washed first with solution of sal-ammoniac, and then with alcohol, dried, and ignited.

The mixture of platinum and iridium obtained by the two ignitions is weighed, and digested in aqua-regia diluted with five times its weight of water, at a temperature of about 110° to 120° Fahr. The aqua-regia is renewed until no longer coloured. The residue is pure *iridium*. On subtracting its weight from that of the mixture, we find the *platinum*.

4. The liquid remaining in No. 3, after separation

of platinum and iridium, is evaporated to expel alcohol, and mixed with nitric acid in excess to destroy the salammoniac, and evaporated almost to dryness. It is now placed in a covered porcelain crucible, and weighed with great care. When dry it is moistened with strong hydrosulphate of ammonia, and dusted over with 30 to 45 grains of pure sulphur. When dry, the crucible is placed within a larger one of clay, the interval between the two being filled up with pieces of charcoal. The two, covered, are set in a cold furnace filled with charcoal, and the fire is lighted at top. The crucibles are raised to a bright red heat, and then allowed to cool. The porcelain crucible now contains sulphides of iron and copper, and metallic palladium, gold, and rhodium. This is digested in a little strong nitric acid at 158° Fahr., which in time dissolves out the iron, copper, and palladium. The solution is poured off, the residue washed by decantation, the solution and washings evaporated to dryness, and strongly ignited. The residue is treated with strong hydrochloric acid, which dissolves the iron and copper. The matter not dissolved is washed by decantation, dried, ignited, and weighed as *palladium*.

The chlorides of iron and copper are evaporated to dryness at a heat little above 212° Fahr., and treated with ammonia. The *iron* remaining undissolved is washed, ignited, and weighed as sesquioxide. The ammoniacal solution is evaporated nearly to dryness, mixed with nitric acid in excess, heated to destroy salammoniac, and the resulting nitrate of copper ignited and weighed as oxide.

5. The residue of No. 4, insoluble in nitric acid, is weighed and treated with very dilute aqua-regia, which takes up the gold, and occasionally a residual trace of platinum. To determine if the latter be present, evaporate to dryness, and redissolve in alcohol and salammoniac. Any residue not taken up is ignited, weighed, and added to the platinum in No. 3. The difference in the weight of the porcelain crucible before and after treatment with aqua-regia gives the weight of the *gold*, from which any trace of platinum found is, of course, deducted.

6. The residue not taken up by aqua-regia is placed in a small porcelain boat, heated in a current of hydrogen gas with the usual precautions, and weighed as metallic *rhodium*.

§ 166. BRONZE AND BRASS.

Substances present—silicium, tin, zinc, iron, copper, and lead.

1. Weigh out about 75 grains, dissolve in pure nitric acid in a beaker covered with an inverted funnel. When dissolved, boil twenty minutes, dilute with twice or thrice its bulk of water, and boil again for about the same time. Filter, wash, dry, ignite, and weigh.

2. *Silica and Oxide of Tin*.—Put this mixture in a porcelain boat, introduce it into a narrow tube, and heat it in a current of hydrogen gas. The tin is reduced, and may then be dissolved in muriatic acid. The *silica* remains, and its weight subtracted from the mixture above, gives the amount of the

oxide of tin, 10 grains of which represent 7.86 grains metallic tin.

3. Evaporate the filtrate of No. 1 to dryness in a platinum capsule, and calcine carefully at a dull red heat. Weigh the residue, triturate in a small mortar, and place about 30 grains in a porcelain tray, and introduce it into a glass tube drawn out at one end to a point, and closed at the other with a cork, which is only used whilst weighing. The tray, tube, and cork are previously weighed together, and the weight of the mixed oxides is determined after they have been heated to dull redness in the apparatus, whilst dry air is drawn through it by the aspirator.

After weighing, a current of pure dry hydrogen gas is passed through the apparatus, and when the common air is expelled, heat is applied by the gas furnace as long as aqueous vapour is deposited in the drawn out end of the tube. The apparatus is cooled in a current of hydrogen gas, and weighed again. The loss of weight shows the amount of oxygen which had been contained in the oxides of iron, lead and copper. Oxide of zinc remains unreduced. If the amount of iron and lead is trifling, the quantity of *copper* present may be ascertained with moderate exactness by multiplying this loss by 4.

4. If the iron and lead cannot be thus overlooked, take pure sulphuric acid (rectified over sulphate of ammonia) diluted with water, in quantity sufficient to dissolve at least double the zinc and iron which appear to be present. Boil this dilute acid to expel air, and let it cool in a bottle (which it should nearly fill) pro-

vided with a stopper or an india-rubber cover. The tray with the oxide of zinc and reduced metals is then put in, and the vessel is frequently shaken. The oxide of zinc and metallic iron soon dissolve. The whole is let settle for some hours, then carefully decanted, and the residue washed with boiled water. Lest any trace of lead or copper may have passed over, a clear solution of HS is added to the liquid, and any brown flakes which are deposited are carefully filtered off and added to the undissolved metals.

5. The solution containing sulphates of zinc and iron is evaporated to dryness, the residue calcined at 752° Fahr., and weighed. Should no iron be present, the weight of zinc is at once known from the weight of anhydrous sulphate found. Should iron be present, proceed as in I. § 29.

6. The mixture of lead and copper, to which any traces of sulphuret thrown down for the solution, No. 4, have been added, is dissolved in sulphuric acid mixed with nitric, the solution evaporated to dryness, and calcined at 752° Fahr. The mixed sulphates are then weighed, the sulphate of copper extracted with water, and the sulphate of lead which remains heated and weighed. Its weight deducted from the total weight of the sulphates gives of course the weight of sulphate of copper.

For confirmation the copper in solution may be determined as in II. § 152.

N.B.—8.02 grains anhydrous sulphate of zinc represent 4.02 oxide of zinc, or 3.22 metallic zinc;

8 grains anhydrous sulphate of copper represent 3·2 metallic copper.—*St. Clair Deville*.

See also I. §§ 74, 75, and 76.

§ 167. PEWTER.

Dissolve in nitro-muriatic acid, using as little as possible of the former. Separate—as directed in Part I.—tin, antimony, bismuth, copper, and lead. Or, dissolve in concentrated muriatic acid, adding by degrees small quantities of nitric. Or, boil in muriatic acid, and add a hot saturated solution of chlorate of potash until the metal dissolves. Tin and lead generally predominate.

§ 168. GERMAN SILVER.

See I. § 75. If lead and iron be present, the former is separated from the copper, and the latter from the nickel and zinc by the methods laid down in Part I.

§ 169. TYPE METAL.

Separate tin and antimony (I. § 86), after dissolving in muriatic acid.

NOTE TO II. § 10.

Bleaching-powder may also be examined by means of a standard solution of protochloride of tin. The value of this tin solution is found by dissolving a known weight of iron in muriatic acid, heating it with nitric acid to convert it into perchloride, and adding a little sulphocyanide of potassium to colour. Ascertain how many degrees of the tin

solution are required to discharge the red colour. A measured quantity of the tin solution is now treated with the solution of a given amount of the chloride of lime, and the amount of tin remaining unconverted into a per-salt, is determined by means of the iron solution. The amount of tin perchlorinized, shows the amount of available chlorine in the sample.

NOTE TO II. § 20.

The determination of oxalic acid is based on the same reaction as that of lime (I. § 10). The oxalic acid is neutralized with ammonia and chloride of calcium added in solution. Proceed as directed for lime, converting the precipitate of oxalate of lime into carbonate of lime by ignition. In this, determine carbonic acid as in I. § 8, *b*; 22 parts of carbonic acid correspond to 36 of oxalic acid. If a free mineral acid be present, add acetate of potash, and precipitate with a mixture of chloride of calcium and acetate of potash.

APPENDIX.

CHEMICAL TABLES.

I.

TABLE OF EQUIVALENTS (HYDROGEN = 1).

Aluminium	13·7	Molybdenum	48·0
Antimony	129·0	Nickel	29·6
Arsenic	75·2	Nitrogen	14·0
Barium	68·6	Osmium	99·6
Bismuth	106·4	Oxygen	8·0
Boron	10·8	Palladium	53·4
Bromine	78·4	Phosphorus	31·4
Cadmium	55·8	Platinum	98·7
Calcium	20·5	Potassium	39·2
Carbon	6·0	Rhodium	52·1
Cerium	46·3	Ruthenium	51·7
Chlorine	35·4	Selenium	40·0
Chromium	28·1	Silicon	14·8
Cobalt	29·6	Silver	108·1
Copper	31·8	Sodium	23·2
Fluorine	18·7	Strontium	44·0
Glucium	17·7	Sulphur	16·0
Gold	199·0	Tantalum	185·0
Hydrogen	1·0	Tellurium	64·0
Iodine	126·0	Thorium	59·6
Iridium	98·7	Tin	59·0
Iron	27·2	Titanium	24·5
Lantanum	36·1	Tungsten	95·0
Lead	103·8	Uranium	217·0
Lithium	6·4	Vanadium	68·6
Magnesium	12·7	Yttrium	32·2
Manganese	27·6	Zinc	32·2
Mercury	101·4	Zirconium	22·4

II.

BOILING POINTS OF SALINE SOLUTIONS.

By means of the following solutions used in the water-bath, any desired temperature may be maintained up to 419° Fahr. :—

Chloride of calcium (10 to 41 per cent.)	212° to 230°
Common salt	up to 230
Oxalic acid	234
Sal ammoniac	238
Carbonate of potash	284
Potash	312
Nitrate of ammonia	356
Soda	419

Higher temperatures are obtained by the oil-bath or hot-air bath.

III.

HYDROMETER TABLES.

1. Table for comparing Beaumè's hydrometer with direct specific gravity—

a. Liquids heavier than water.

0	=	1.000	39	=	1.373
3	"	1.020	42	"	1.414
6	"	1.040	45	"	1.455
9	"	1.064	48	"	1.500
12	"	1.089	51	"	1.547
15	"	1.114	54	"	1.594
18	"	1.140	57	"	1.659
21	"	1.170	60	"	1.717
24	"	1.200	63	"	1.779
27	"	1.230	66	"	1.848
30	"	1.261	69	"	1.920
33	"	1.295	72	"	2.000
36	"	1.333			

b. Liquids lighter than water.

10	=	1.000	26	=	.892
11	"	.990	27	"	.886
12	"	.985	28	"	.880
13	"	.977	29	"	.874
14	"	.970	30	"	.867
15	"	.963	31	"	.861
16	"	.955	32	"	.856
17	"	.949	33	"	.852
18	"	.942	34	"	.847
19	"	.935	35	"	.842
20	"	.928	36	"	.837
21	"	.922	37	"	.832
22	"	.915	38	"	.827
23	"	.909	39	"	.822
24	"	.903	40	"	.817
25	"	.897			

2. Table for comparing Twaddel with Beaumè.

Beaumè.	Twaddel.	Beaumè.	Twaddel.
0	= 0	39	= 74
3	" 4	42	" 83
5	" 8	43	" 91
9	" 13	48	" 100
12	" 18	51	" 109
15	" 23	54	" 119
18	" 28	57	" 132
21	" 34	60	" 143
24	" 40	63	" 156
27	" 47	66	" 168
30	" 52	69	" 184
33	" 59	72	" 200
36	" 66		

3. To convert Twaddell's scale into direct specific gravity—

Multiply by 5, and add 1·000.

Specific gravity into Twaddell—

Subtract 1·000, and divide by 5.

IV.

RULE FOR CONVERTING CENTIGRADE INTO FAHRENHEIT.

If the temperature be above the freezing-point of water (0° cent.), multiply by 9, divide by 5, and add 32 to the quotient.

If the temperature be below 0° cent., but above 18° cent., multiply by 9, divide by 5, and subtract the result from 32.

If below 18° cent., multiply by 9, divide by 5, and subtract 32 from the result.

TO CONVERT FAHRENHEIT INTO CENTIGRADE.

If above 32° Fahr., subtract 32, multiply by 5, and divide by 9.

If below 32° Fahr., but above 0° Fahr., subtract the temperature from 32, multiply by 5, and divide by 9.

If below 0° Fahr., add the temperature to 32, multiply by 5, and divide by 9.

The conversion of Reaumur's scale into Fahrenheit, and *vice versa* is effected in the same manner, the number 4 being used instead of 5 as a multiplier or divisor.

V.

DECIMAL WEIGHTS AND MEASURES.

1 milligramme	=	0·01544 grain.
1 centigramme	"	0·15442 grain.
1 decigramme	"	1·54424 grains.
1 gramme	"	15·44242 grains.
1 kilogramme	"	2·67951 lb. troy.
"	"	2·20486 lb. avoirdupois.
1 cubic centimetre	"	0·06103 cubic inch.
"	"	15·4 grain measures.
1 litre	"	0·22017 gallon.
"	"	0·88066 quart.
1 stere	"	220·16643 gallons.

VI.—TABLE OF THE QUANTITY OF OIL OF VITROIL, OF SPECIFIC GRAVITY 1·8485, AND OF ANHYDROUS ACID, IN 100 PARTS OF DILUTE SULPHURIC ACID, AT DIFFERENT DENSITIES.

Liquid.	Specific Gravity.	Dry.	Liquid.	Specific Gravity.	Dry.
100	1·8485	81·54	50	1·3884	40·77
99	1·8475	80·72	49	1·3788	39·95
98	1·8460	79·90	48	1·3697	39·14
97	1·8439	79·09	47	1·3612	38·32
96	1·8410	78·28	46	1·3530	37·51
95	1·8376	77·46	45	1·3440	36·69
94	1·8336	76·65	44	1·3345	35·88
93	1·8290	75·83	43	1·3255	35·06
92	1·8233	75·02	42	1·3165	34·25
91	1·8179	74·20	41	1·3080	33·43
90	1·8115	73·39	40	1·2999	32·61
89	1·8043	72·57	39	1·2913	31·80
88	1·7962	71·75	38	1·2826	30·98
87	1·7870	70·94	37	1·2740	30·17
86	1·7774	70·12	36	1·2654	29·35
85	1·7673	69·31	35	1·2572	28·54
84	1·7570	68·49	34	1·2490	27·72
83	1·7465	67·68	33	1·2409	26·91
82	1·7360	66·86	32	1·2334	26·09
81	1·7245	66·05	31	1·2260	25·28
80	1·7120	65·23	30	1·2184	24·46
79	1·6993	64·42	29	1·2108	23·65
78	1·6870	63·60	28	1·2032	22·83
77	1·6750	62·78	27	1·1956	22·01
76	1·6630	61·97	26	1·1876	21·20
75	1·6520	61·15	25	1·1792	20·38
74	1·6415	60·34	24	1·1706	19·57
73	1·6321	59·52	23	1·1626	18·75
72	1·6204	58·71	22	1·1549	17·94
71	1·6090	57·89	21	1·1480	17·12
70	1·5975	57·08	20	1·1410	16·31
69	1·5868	56·26	19	1·1330	15·49
68	1·5760	55·45	18	1·1246	14·68
67	1·5648	54·63	17	1·1165	13·86
66	1·5503	53·82	16	1·1090	13·05
65	1·5390	53·00	15	1·1019	12·23
64	1·5280	52·18	14	1·0953	11·41
63	1·5170	51·37	13	1·0887	10·60
62	1·5066	50·55	12	1·0809	9·78
61	1·4960	49·74	11	1·0743	8·97
60	1·4860	48·92	10	1·0682	8·15
59	1·4760	48·11	9	1·0614	7·34
58	1·4660	47·29	8	1·0544	6·52
57	1·4560	46·48	7	1·0477	5·71
56	1·4460	45·66	6	1·0405	4·89
55	1·4360	44·85	5	1·0336	4·08
54	1·4265	44·03	4	1·0268	3·26
53	1·4170	43·22	3	1·0206	2·446
52	1·4073	42·40	2	1·0140	1·63
51	1·3977	41·58	1	1·0074	0·8154

VII.—TABLE OF THE QUANTITY OF DRY NITRIC ACID IN 100 PARTS OF LIQUID ACID, AT DIFFERENT DENSITIES.

Specific Gravity.	Real acid in 100 parts of the Liquid.	Specific Gravity.	Real acid in 100 parts of the Liquid.
1·5000	79·700	1·2947	39·850
1·4980	78·903	1·2887	39·053
1·4960	78·106	1·2826	38·256
1·4940	77·309	1·2765	37·459
1·4910	76·512	1·2705	36·662
1·4880	75·715	1·2644	35·865
1·4850	74·918	1·2583	35·068
1·4820	74·121	1·2523	34·271
1·4790	73·324	1·2462	33·474
1·4760	72·527	1·2402	32·677
1·4730	71·730	1·2341	31·880
1·4700	70·933	1·2277	31·083
1·4670	70·136	1·2212	30·286
1·4640	69·339	1·2148	29·489
1·4600	68·542	1·2084	28·692
1·4570	67·745	1·2019	27·895
1·4530	66·948	1·1958	27·098
1·4500	66·155	1·1895	26·301
1·4460	65·354	1·1833	25·504
1·4424	64·557	1·1770	24·707
1·4385	63·760	1·1709	23·910
1·4346	62·963	1·1648	23·113
1·4306	62·166	1·1587	22·316
1·4269	61·369	1·1526	21·519
1·4228	60·572	1·1465	20·722
1·4189	59·775	1·1403	19·925
1·4147	58·978	1·1345	19·128
1·4107	58·181	1·1286	18·331
1·4065	57·384	1·1227	17·534
1·4023	56·587	1·1168	16·737
1·3978	55·790	1·1109	15·940
1·3945	54·993	1·1051	15·143
1·3882	54·196	1·0993	14·346
1·3833	53·399	1·0935	13·549
1·3783	52·602	1·0878	12·752
1·3732	51·805	1·0821	11·955
1·3681	51·068	1·0764	11·158
1·3630	50·211	1·0708	10·361
1·3579	49·414	1·0651	9·564
1·3529	48·617	1·0595	8·767
1·3477	47·820	1·0540	7·970
1·3427	47·023	1·0485	7·173
1·3376	46·226	1·0430	6·376
1·3323	45·429	1·0375	5·579
1·3270	44·632	1·0320	4·782
1·3216	43·835	1·0267	3·985
1·3163	43·038	1·0212	3·188
1·3110	42·241	1·0159	2·391
1·3056	41·444	1·0106	1·594
1·3001	40·647	1·0053	0·797

VIII.

TABLE OF THE QUANTITY OF REAL HYDROCHLORIC ACID IN THE
LIQUID ACID OF DIFFERENT SPECIFIC GRAVITIES.

Acid of 120 in 100.		Specific Gravity.		Chlorine.		Hydrochloric Gas.
100	1·2000	39·675	40·777
99	1·1982	39·278	40·369
98	1·1964	38·882	39·961
97	1·1946	38·485	39·554
96	1·1928	38·089	39·146
95	1·1910	37·692	38·738
94	..	1·1893	37·296	38·330
93	1·1875	36·900	37·923
92	1·1857	36·503	37·516
91	1·1846	36·107	37·108
90	1·1822	35·707	36·700
89	1·1802	35·310	36·292
88	1·1782	34·913	35·884
87	1·1762	34·517	35·476
86	1·1741	34·121	35·068
85	1·1721	33·724	34·660
84	1·1701	33·328	34·252
83	1·1681	32·931	33·845
82	1·1661	32·535	33·437
81	1·1641	32·136	33·029
80	1·1620	31·743	32·621
79	1·1599	31·343	32·213
78	1·1578	30·946	31·805
77	1·1557	30·550	31·398
76	1·1536	30·153	30·990
75	1·1515	29·757	30·582
74	1·1494	29·361	30·174
73	1·1473	28·964	29·767
72	1·1452	28·567	29·359
71	1·1431	28·171	28·951
70	1·1410	27·772	28·544
69	1·1389	27·376	28·136
68	1·1369	26·979	27·728
67	1·1349	26·583	27·321
66	1·1328	26·186	26·913
65	1·1308	25·789	26·505
64	1·1287	25·392	26·098
63	1·1267	24·996	25·690
62	1·1247	24·599	25·282
61	1·1226	24·202	24·874
60	1·1206	..	23·805	24·466
59	1·1185	23·408	24·058
58	1·1164	23·012	23·650
57	1·1143	22·615	23·242
56	1·1123	22·218	22·834
55	1·1102	21·822	22·426
54	1·1082	21·425	22·019
53	1·1061	21·028	21·611
52	1·1041	20·632	21·203

Acid of 120 in 100.	Specific Gravity.	Chlorine.	Hydrochloric Gas.
51	1·1020	20·235	20·796
50	1·1000	19·837	20·388
49	1·0980	19·440	19·980
48	1·0960	19·044	19·572
47	1·0939	18·647	19·165
46	1·0919	18·250	18·757
45	1·0899	17·854	18·349
44	1·0879	17·457	17·941
43	1·0859	17·060	17·534
42	1·0838	16·664	17·126
41	1·0818	16·267	16·718
40	1·0798	15·870	16·310
39	1·0778	15·474	15·902
38	1·0758	15·077	15·494
37	1·0738	14·680	15·087
36	1·0718	14·284	14·679
35	1·0697	13·887	14·271
34	1·0677	13·490	13·863
33	1·0657	13·094	13·456
32	1·0637	12·597	13·049
31	1·0617	12·300	12·641
30	1·0597	11·903	12·233
29	1·0577	11·506	11·825
28	1·0557	11·109	11·418
27	1·0537	10·712	11·010
26	1·0517	10·316	10·602
25	1·0497	9·919	10·194
24	1·0477	9·522	9·786
23	1·0457	9·126	9·379
22	1·0437	8·729	8·971
21	1·0417	8·332	8·563
20	1·0397	7·935	8·155
19	1·0377	7·538	7·747
18	1·0357	7·141	7·340
17	1·0337	6·745	6·932
16	1·0318	6·348	6·524
15	1·0298	5·951	6·116
14	1·0279	5·554	5·709
13	1·0259	5·158	5·301
12	1·0239	4·762	4·893
11	1·0220	4·365	4·486
10	1·0200	3·998	4·078
9	1·0180	3·571	3·670
8	1·0160	3·174	3·262
7	1·0140	2·778	2·854
6	1·0120	2·381	2·447
5	1·0110	1·984	2·039
4	1·0080	1·588	1·631
3	1·0060	1·191	1·224
2	1·0040	0·795	0·816
1	1·0020	0·397	0·408

BASIC COMPOUNDS, ACIDS, AND ORGANIC ACIDS.

The following table shows the precipitates produced by certain reagents in the solutions of the various earths, metallic oxides, acids, &c. The figure 1 below the colour of the precipitate signifies that it is soluble in an excess of the precipitant; 2, insoluble; 3, that the precipitate only appears after prolonged standing.

Where the space is left blank, no precipitate or other marked change is produced.

Both basic oxides and acids are supposed to be in combination, and neutralized where a neutral soluble compound exists. For blow-pipe tests, the reader is referred to Dr. Muspratt's edition of "Plattner on the Blow-pipe."

A. Basic Compounds.	Potash.	Ammonia.	Carbonate of Soda.	Carbonate of Ammonia.	Chromate of Potash.	Ferrocyanide of Potassium.	Ferricyanide of Potassium.	Remarks.
ALUMINA.....	White. 1.	White. 2.	White. 2.	White. 2.	...	White. 3. turns green.	...	Precipitated by phosphate and tungstate of soda and succinate of ammonia.
ANTIMONY.....	White. 1.	White. 2.	White.	White.	...	White. insoluble in muriatic acid.	...	Muriatic solution precipitated by excess of water.
BARIUM.....	White.	...	White. 2.	White. 2.	Yellow. 2.	Whitish yellow. 3.	...	White insoluble precipitate with solution of sulphate of lime.
BISMUTH	White. 2.	White. 2.	White. 2.	White. 2.	Yellow. 2. soluble in nitric acid.	White.	Yellow.	Nitric acid, solution precipitated by excess of water.

CADMIUM.....	White. 2.	White. 1.	White. 2.	White. 2.	...	White.	Yellow.	...
CALCIUM	White. 2.	White. 2.	...	White. 3. (strong sol.)	...	White precipitate with oxalate of ammonia; precipitate with tungstate of soda.
CERIUM.....	White. 2.	White. 2.	White. 1.	White. 1.	...	White.
CHROMIUM ...	Green. 1.	Green. 2.	Green.	Green.	Yellow-brown.	Green.
COBALT	Blue, pale red if boiled 2.	Blue. 1.	Red. 2.	Red. 2.	...	Green.	Brown-red.	Oxalic acid, a rose precipitate.
COPPER.....	Blue. 2. black if boiled.	Green. 1. sol. blue.	Blue. 2. black if boiled.	Light green. 1. sol. blue.	Red-brown.	Red-brown.	Green-yellow.	Tincture of guaiacum, a blue-green colour.
DIDYMIUM ...	Violet.	Violet.
GLUCINUM	White. 1.	White. 2.	White. 1.	White. 1.	...	White. 3.	...	Phosphate soda, a white precipitate. Tincture of galls, a yellow precipitate.

BASIC COMPOUNDS—continued.

A. Basic Compounds.	Potash	Ammonia.	Carbonate of Soda.	Carbonate of Ammonia.	Chromate of Potash	Ferrocyanide of Potassium	Ferridcyanide of Potassium.	Remarks.
GOLD	Brown-black.	Yellow.	...	Yellow.	Reduced by oxalic acid.
IRIDIUM	Brown-black. sol. heated turns blue. 3.	Brown-black.	Brown-red, redissolves by degrees.	...	Olive green	Deep blue with nitrate of silver; turns colour- less afterwards.
Proto { IRON Sesqui {	White, then green. Red-brown.	White, then green. Red-brown.	White, then green. Red-brown.	White, then green. Red-brown.	Yellow- brown. Brown.	White. Blue.	Blue. Sulpho-cyanide of potas- sium, a red colour.
LANTANUM ...	White. 2.	White. 2.	White. 2.	White. 2.
LEAD	White. 1.	White. 2.	White. 2.	White. 2.	Yellow. 2.	White.	...	Yellow precipitate with iodide of potassium, soluble in excess.
LITHIA	White. in strong sols.	White. in strong sols.

MAGNESIA	White.	...	White.	White.	Crystalline precipitate with phosphate of soda and ammonia.
MANGANESE...	White, then brown.	White, then brown	White.	White.	Dark brown 3.	White.	Brown.	...
MERCURY (sub-salts).	Black.	Black.	Grey.	Grey.	Red.	White.	Red-brown, then white.	...
MERCURY (proto-salts).	Orange. 2.	White. 2.	Reddish-brown. 2.	White. 2.	Orange.	White.	Yellow (except in chloride).	Scarlet with iodide of potassium; soluble in excess of either liquid.
MOLYBDENUM	Brown-black. 2.	Brown-black.	Brown. 1.	Brown. 1.	Yellow-grey.	Red-brown.	Red-brown.	...
NICKEL	Apple green 2.	Green. 1. sol. violet.	Apple-green. 2.	Apple green 1. sol. green.	Reddish-yellow.	White.	Greenish-yellow.	...
OSMIUM	Black on heating.	Brown. 3.	Brown. 3.	Brown. 3.
PALLADIUM...	Brown-yellow. 1.	Flesh-coloured (only in chloride).	Brown. 1.	Flesh-coloured (in chloride)	...	White jelly. 3.	White jelly.	Yellow-white with cyanide of mercury.

BASIC COMPOUNDS—continued.

A. Basic Compounds	Potash.	Ammonia.	Carbonate of Soda.	Carbuaate of Ammonia.	Chromate of Potash.	Ferrocyanide of Potassium.	Ferridcyanide of Potassium	Remarks.
PLATINUM	Yellow. 1.	Yellow.	Yellow. 2.	Yellow.	Deep red.	Brown-red precipitate with iodide of potassium; red colour in dilute solutions.
POTASH	Yellow precipitate with bichloride of platinum.
RHODIUM.....	Yellow, brown on boiling.	Yellow.	Yellow. 3.	Yellow.	Green vitriol gives a yellow precipitate.
RUTHENIUM...	Black- brown. 1.	Black- brown.	Black- brown.	Black- brown.	Black precipitate with nitrate of silver turns white; clear liquid rose coloured.
SILVER	Brown. 2.	Brown. 1.	White.	White. 1.	Red.	White.	Brown-red.	White curd with muriatic acid.
SODA
STREONTIA	White.	...	White.	White.	Yellow.

THIORINA	White. 1.	White.	White.	...	White.
TIN (proto) ...	Cream- white. 1.	White. 2.	White.	Greenish- white.	White.	White.	Purple brown precipi- tate with chloride of gold.
TIN (per)	White. 1.	White. 1.	White. 2.	Yellow.	Yellow- white jelly. 3.	...	No precipitate with gold; sulphuric acid a white turbidity in very dilute solution.
URANIUM	Brown. 2.	Dirty green. 1.	Dirty green.	...	Brown-red.	Brown-red. 3.	Yellow granular precipi- tate with sulphate of ammonia on boiling.
VANADIUM ..	Grey. 1.	Grey. 1.	Yellow, turns green.	Green.	Blue-black with tincture of galls.
YTTRIA	White. 2.	White. 2.	White. 1.	...	White.
ZINC	White. 1.	White. 2.	White. 1.	Yellow, (soluble in ammonia.)	White.	Orange- yellow.	...
ZIRCONIA	White. 2.	White. 1.	White. 1.	...	White.

ACIDS.

B. Acids.	Chloride of Barium.	Nitrate of Silver.	Nitrate of Lime.	Nitrate of Lead.	In fusion of Galls.	Hs.	Nitrate of suboxide of mercury.	Remarks.
ANTIMONIC....	White.	White.	Orange.	Orange-yellow.	Insoluble in water.
ANTIMONIOUS	White.	White.	Orange.
ARSENIC	White.	Red brown.	Yellow; soluble in alkalies.	White; turns red when dry.	...
ARSENIOUS ...	White.	Pale yellow.	Yellow; soluble in alkalies.	White.	...
BORACIC	White.	White.	White.	Brown.	Tinges flame of alcohol green; brown-red precipitate with chlo- ride of mercury.
BROMIC.....	White.	White.	...	White.	Yellowish- white.	Sulphuric acid liberates bromine.

CARBONIC.....	White.	White.	White.	White.	Decomposed by acids with effervescence.
CHLORIC.....	Sulphuric acid disengages greenish-yellow vapours.
CHLOROUS.....	...	Black.	...	White, then yellow.
CHROMIC.....	Yellow.	Deep red.	Slight yellow in very strong solution.	Yellow.	...	Green liquid; precipitate of sulphur.	Bright red. Turns green if heated with muriatic acid and sugar.
HYDROBROMIC.....	...	Yellow.	...	White.	Gives off bromine when heated with nitric acid.
HYDROCHLORIC.....	...	White, soluble in ammonia.	...	White in strong solutions.	Gives off chlorine when heated with red lead.
HYDROCYANIC	...	White, soluble in cyanide of potassium.	...	White.	Add persulphuret of ammonium, and heat in a watch-glass; perchloride of iron then gives a blood-red colour.

ACIDS—continued.

B. Acids.	Chloride of Barium.	Nitrate of Silver.	Nitrate of Lime.	Nitrate of Lead.	Infusion of Galls.	H.S.	Nitrate of Suboxide of Mercury.	Remarks.
HYDRIODIC....	...	Pale yellow.	...	Yellow. 1.	Greenish-yellow; chloride of mercury scarlet.	Hyponitric acid and starch paste give a purple tint.
HYDROFLUORIC.....	White.	...	White; gelatinous.	White.	Sulphuric acid with heat gives off fumes that corrode glass.
HYDROSELENIC.....	Reddens organic matter; peculiar scent when heated.
HYDROSULPHURIC	Black.	...	Black.	Black.	Gives off H.S. when mixed with dilute sulphuric.
HYPOSULPHURIC	Smell of sulphurous acid when heated; sulphate remains.
HYPOSULPHUROUS.....	...	White, then black brown.	...	White; black at 212° Fahr.	Black on boiling.	...

IODIC.....	White; in strong solution.	White.	White; in strong solution.	White.	White.	Converted into iodide by heat.
MANGANIC.....	...	Black.	Black.	Solution changes from green to red with acids.
MOLYBDIC	White.	White.	White.	White.	Green with muriatic acid.	Brown; green liquid above.	White.	Blue with chloride of tin.
NITRIC.....	Pour the solution upon strong sulphuric acid and add a crystal of green vitriol; a red colour.
PHOSPHORIC...	White.	Yellow.	White.	White.	White crystalline.	Yellow precipitate with molybdate of am- monia and nitric acid.
PHOSPHOROUS	White.	White, then brown.	White.	White.
SELENIC	White; insoluble in acids.	White.	White.	White.	...	Lemon-yel- low if boiled with muri- atic acid previously.	...	Sulphate of indigo is decolourized by boil- ing with seleniates and muriatic acid.

ACIDS—continued.

L. Acids.	Chloride of Barium	Nitrate of Silver.	Nitrate of Lime	Nitrate of Lead.	Infusion of Gal's	Hs.	Nitrate of Suboxide of Mercury.	Remarks.
SELENIOS.....	White.	White.	White.	White.	...	Lemon-yellow; red on drying.	White.	Zinc gives a red-brown precipitate.
SILICIC	White.	Yellow.	White.	White.	Insoluble in acids after ignition.
SULPHURIC ...	White; insoluble in acids.	White.	White.	White.
SULPHUROUS..	White.	White.	White.	White.	Turns chromic; acid green.
TANTALIC	Orange-yellow colour.	Precipitated by muriatic acid from its alkaline solution.
TELLURIC	White.	Light brown. 3.	Yellowish-brown.	Mixed muriatic and sulphurous acids give a black precipitate.
TELLUROUS...	Brown.	Brown.	Deep yellow turning brown.	Zinc gives a black precipitate.

TITANIC	Reddish precipitate if no excess of muriatic ammonia in acid present. ...	White with hydrosulphate of ammonia in excess.	...	Zinc gives a blue colour in a muriatic solution of an alkaline titanate.
TUNGSTIC	White.	White.	White.	White.	Brown precipitate with hydro-sulphate of ammonia.	Yellow.	Zinc gives a blue colour in the muriatic precipitate of alkaline tungstates.
VANADIC	Yellow, turning white.	Yellow.	...	Yellow, turning white.	Blue in solutions of vanadic acid in acids.	Dark yellow; redissolves spontaneously.	...

ORGANIC ACIDS.

Organic Acids.	Chloride of Barium.	Nitrate of Silver.	Nitrate of Lime.	Nitrate of Lead.	Infusion of Galls.	H ₂ S.	Nitrate of Sesquioxide of Mercury.	Remarks.
ACETIC	White crystalline.	Blood-red, with neutral per-salts of iron; muriatic destroys this colour.
BENZOIC	White.	Brown-yellow precipitate with neutral per-salts of iron.
CITRIC	White on boiling.	White.	White.	White.	White.	...
FORMIC	White, then black.
LACTIC	White.
MALIC	White, then grey.	White with alcohol.	White.	White.	...
SUCCINIC	White.	...	White.	White.	Red-brown precipitate with chloride of iron.

TARTARIC	White.	White.	White, soluble in excess of potash.	White, soluble in ammonia.	White.	Gives a white crystalline precipitate with chloride of potassium and alcohol.
URIC	White.	White.	White.	White.	Solution in nitric acid leaves purple-red residue on evaporation.
OXALIC	White.	White.	White.	White.	White.	White precipitate with solution of sulphate of lime.

OXIDES PRECIPITABLE AS SULPHURETS BY HS FROM
ACID SOLUTIONS.

	Colour.	Soluble in
Antimony.....	Orange	Potash, sulphuret of potassium, and muriatic.
Arsenic.....	Yellow	Potash, ammonia, hydrosulph. ammonia.
Bismuth.....	Black	Nitric.
Cadmium.....	Yellow	Nitric, muriatic.
Copper.....	Black	Nitric, muriatic, cyanide potassium.
Gold.....	Black	Nitro-muriatic, hydrosulph. ammonia.
Iridium.....	Brown	Nitric.
Lead.....	Black	Nitric, muriatic, and acetic, if heated.
Mercury.....	Black	Potash, nitro-muriatic.
Molybdenum....	Black-brown..	Hydrosulph. ammonia, nitric, and sulphuric.
Osmium.....	Brown-yellow..	Nitric.
Palladium.....	Black	Muriatic.
Platinum.....	Black	Nitro-muriatic, hydrosulph. ammonia.
Rhodium.....	Brown	Nitric.
Ruthenium.....	Yellow	Nitric.
Silver.....	Black	Sulphuric, nitric (boiling).
Tellurium.....	Brown	Potash, ammonia.
Tin.....	{ Brown	Potash.
(Selenious acid) ..		Ammonia, potash.
	Orange-red.	

SUBSTANCES WHICH CAUSE A MILKY PRECIPITATE OF SULPHUR WHEN
THEIR ACID SOLUTION IS TREATED WITH HS.

Chromic acid.
 Ferric oxide.
 Manganic sesquioxide and acid.
 Permanganic acid.
 Bromic acid.
 Chloric acid.
 Iodic acid.
 Hyposulphuric acid.
 Sulphurous acid.

Oxides thrown down by HS are also thrown down from concentrated hot acid solutions by the hyposulphite of soda, which is best introduced, not in solution, but in fragments. Bismuth does not, as commonly stated, form an exception.

OXIDES PRECIPITABLE AS SULPHURETS BY HYDROSULPHATE OF AMMONIA
FROM NEUTRAL OR ALKALINE SOLUTIONS.

Antimony	Orange.....	Soluble in excess.
Arsenic.....	Yellow.....	Soluble in excess.
Bismuth	Brown-black.	
Cadmium.....	Yellow.	
Cobalt	Black.	
Copper.....	Black.....	Soluble in excess (slightly if deficient in sulphur).
Gold	Brown-black...	Soluble in excess.
Iridium	Brown.....	Soluble in excess.
Iron.....	Black.	
Lead	Black.	
Manganese.....	Flesh-colour.	
Mercury.....	Black.	
Molybdenum ...	Yellow-brown..	Soluble in excess.
Nickel.....	Black.....	Soluble if hydrosulphate, deficient in sulphur.
Osmium	Yellow-brown.	
Palladium	Black.	
Platinum	Brown-black....	Soluble in large excess.
Ruthenium.....	Brown-black.	
Rhodium.....	Brown.	
Silver	Black.	
Tin	{ Brown.	
	{ Yellow.....	Soluble in excess.
Tellurium.....	Deep brown....	Soluble in excess.
Tungsten.....	Brown.....	Soluble in excess.
Uranium.....	Red-brown.	
Vanadium.....	Brown-black....	Soluble in excess.
Zinc	White.....	Insoluble in potash.
(Selenious acid) ..	Yellow.....	Soluble in excess.

The following are thrown down by hydrosulphate of ammonia as hydrous oxides :—

Alumina	White.....	Soluble in potash.
Cerium	White.	
Chrome	Pale green.	
Didymium.....	(Red basic salt.)	
Glucinum.....	White.	
Lanthanum	White.	
Titanium	White.	
Thorium.....	White.	
Yttrium.....	White.	
Zirconium.....	White.	

OXIDES SOLUBLE IN AMMONIA.

Antimony (antimonious and antimonie acids).
 Arsenic.
 Cadmium.
 Cobalt (in presence of ammoniacal salts, or with access of air).
 Copper.
 Iridium (imperfectly).
 Iron (protoxide).
 Manganese (in presence of free acid and ammoniacal salts).
 Nickel.
 Osmium (peroxide only).
 Palladium.
 Ruthenium (imperfectly).
 Silver.
 Tellurium.
 Tin (peroxide).
 Tungsten (acid).
 Zinc.

The action of alkalies towards the following is interfered with by—

AMMONIACAL SALTS.

Cobalt.		Molybdenum.
Copper (except potash in large excess).		Nickel.
Iron, protoxide.		Silver.
Magnesia.		Uranium.
Manganese.		Zinc.

ORGANIC MATTER.

Alumina.		Manganese.
Antimony.		Mercury.
Cadmium.		Nickel.
Cerium.		Platinum.
Cobalt.		Tin.
Copper.		Titanium.
Glucina.		Silver.
Iron.		Uranium.
Magnesia.		Zinc.

BEHAVIOUR OF SOLUBLE SALTS OF THE METALS, ETC., WITH A SOLUTION
OF CYANIDE OF POTASSIUM.

Compound of	Colour of precipitate, &c., or other	Sol. in an excess of cold cyanide.
Cadmium.....	Yellow-white, gelatinous precipitate.	
Cobalt.....	Cinnamon colour precipitate.	
Chrome, protoxide,.....	White precipitate	
“ sesquioxide.....	Green “ } v. sp. sol. in excess.	
Gold, protoxide.....	Yellow, crystalline precipitate.	
“ teroxide.....	Colourless crystals.	
Iron, protoxide.....	Light red-brown precipitate (soluble in large excess).	
“ sesquioxide.....	Red-brown precipitate (hydrous peroxide), partly soluble.	
Manganese, protoxide....	Greyish-yellow (solution brown-red) precipitate.	
Mercury.....	Colourless crystals.	
Silver.....	White curdy precipitate.	
Zinc.....	Snow-white powdery precipitate.	

Nickel.....	Pale apple-green precipitate.	Soluble in excess, hot.
Palladium.....	Pale yellow precipitate.	
Platinum....	Yellow precipitate.	
Uranium.....	Splendid yellow precipitate; sparingly soluble.	

Lead.....	}	White precipitate (cyanide).	} Insoluble in excess, hot or cold.	
Bismuth.....		}		White precipitates, carbonates, or oxides
Barium.....				
Calcium.....				
Strontium.....				
Magnesium.....				
Alumina.....	}	White precipitate as hydrate.		
Tin.....		}		Imperfectly precipitated as oxides.
Antimony.....				
Potassium.....	}	No precipitate.		
Sodium.....				
Lithium.....				
Ammonium.....				

BEHAVIOUR OF INSOLUBLE COMPOUNDS OF THESE METALS TOWARDS
A SOLUTION OF THE CYANIDE.

Cadmium,	} metallic; soluble with access of air.	} Soluble in a <i>cold</i> solution of cyanide.	
Gold,			
Silver,			
Copper,	} metallic; soluble even when not in contact with air.		
Iron,			
Nickel,			
Zinc,			
Copper, protoxide, hydrate.			
" sulphide.			
Cadmium, carbonate.			
Antimony, sulphide (recently precipitated), slowly soluble.			
Cobalt, sulphide (recently precipitated).			
Manganese, sulphide (recently precipitated).			
Mercury, suboxide.			
Silver, chloride, and iodide.			
Zinc, carbonate, and oxide.			

Antimony persulphide.	} recently precipitated.	} Soluble in <i>hot</i> solution of cyanide.
Iron, sulphides,		
Nickel, sulphide,		
Tin, sulphides, partly soluble.		
Zinc, sulphide, recently precipitated.		

Cadmium,	} sulphides of	} Insoluble in excess, hot or cold.	
Mercury,			
Palladium,			
(SH does not precipitate sulphide of palladium from a cyanide solution, except muriatic acid be added.)			
Silver, sulphide (except when precipitated from a very dilute solution).	} carbonates of		
Lead, sulphide.			
Lead,			
Bismuth,			
Baryta,			
Strontia,			
Lime,			
Magnesia,			

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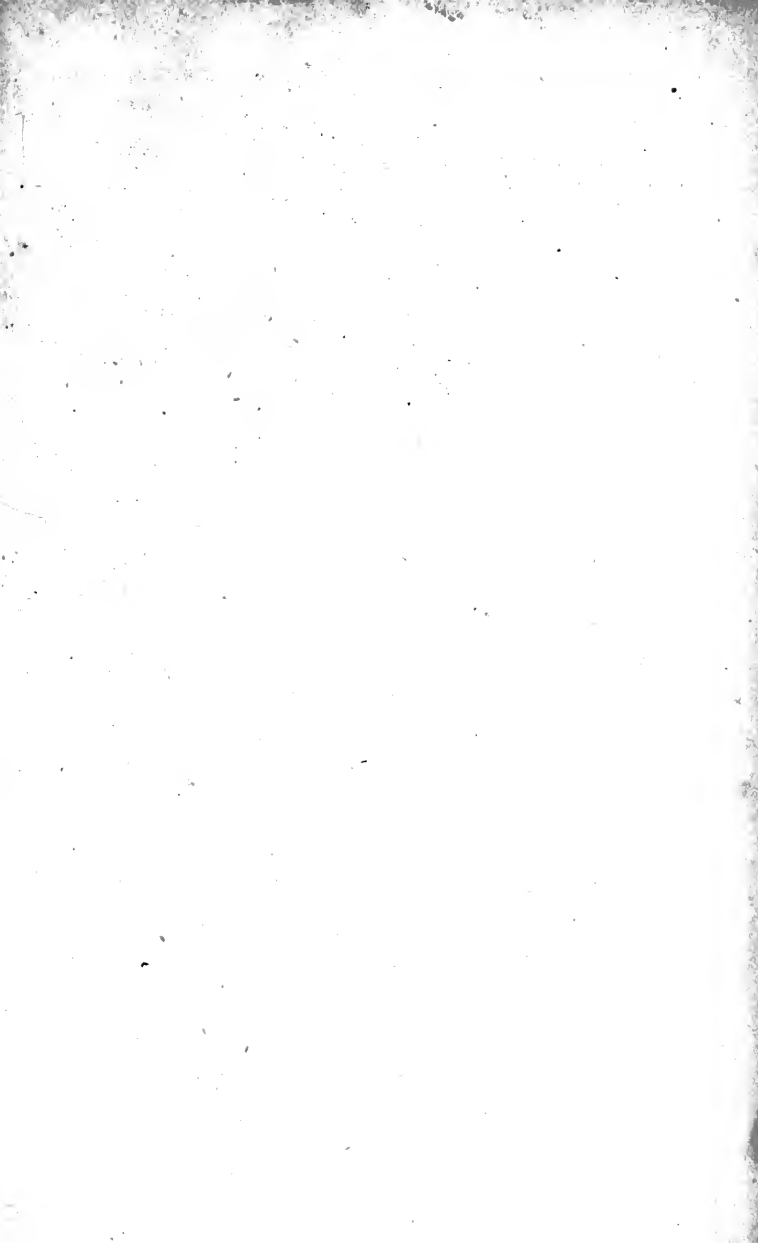
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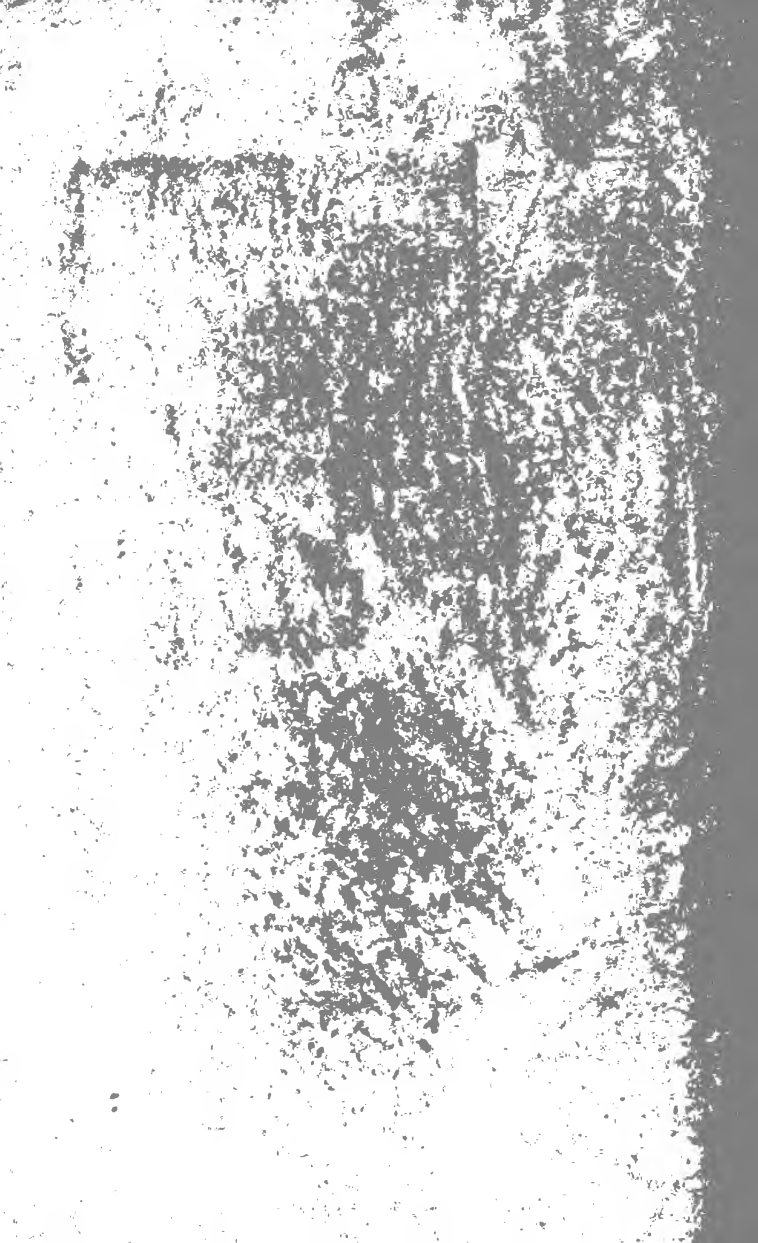
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